Magnétométrie par mesure de susceptibilité AC
-
Magnetometry by AC susceptibility measurements

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What kind of diffent *measurements* can we make with AC susceptibility ?

What kind of *information* can we extract from measurements ?

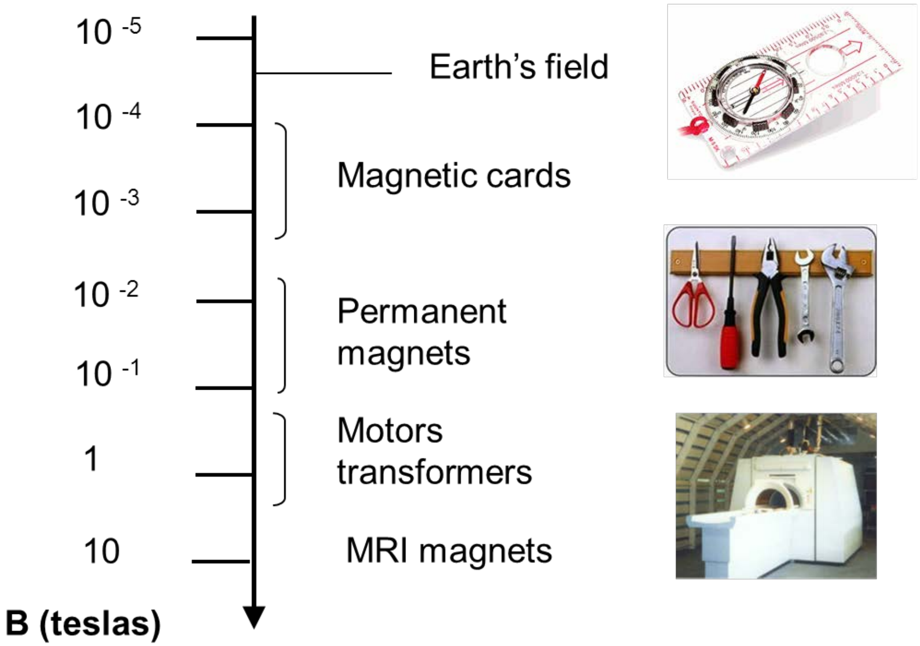
Outline

- What are we measuring?
- How are we measuring?
- What kind of information can we extract?
- Beyond the classic setup : variants and particular designs

What are we talking about ?

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M})$$

H	**= magnetic field**	**[A / m]**
M	**= magnetization**	**[A / m]**
B	**= magnetic induction**	**[T]**



H and M are expressed in the same units

(in Nancy too)

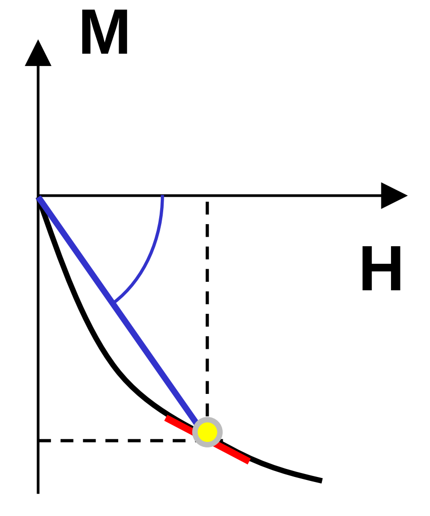
And a little bit more …

m = **magnetic moment** **[A.m²]**

M = **magnetization** **[A / m]**
(= m / V)

χ_{DC} **= magnetic susceptibility**
(= M / H)
[DC]

χ_{AC} **= magnetic susceptibility**
(= dM / dH)
[AC]



And a little bit more …

m = f (physics, applied field, volume)

M = f (physics, applied field, volume)

χ_{DC}, χ_{AC} **= f (physics, applied field, volume)**

So: do not confuse the two m's : « M » and « m »

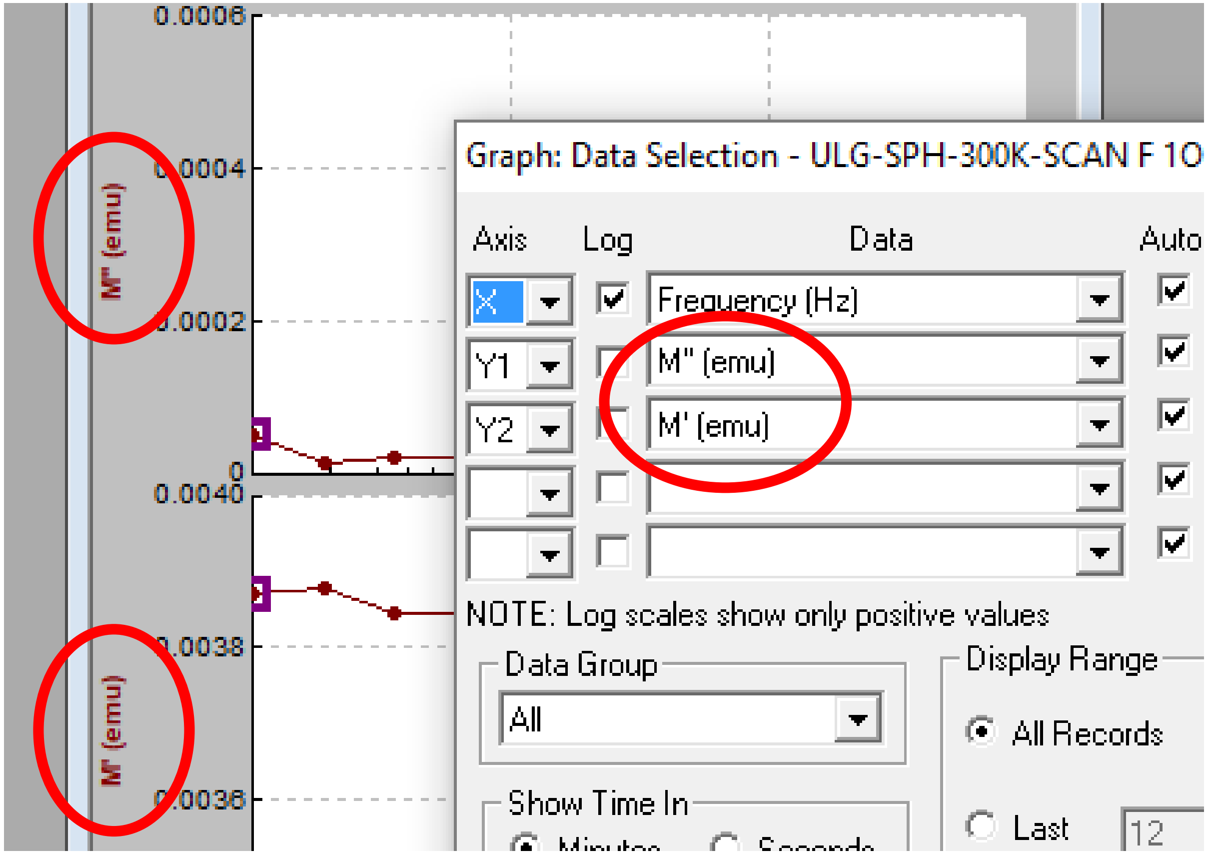
Magnetic moment [Am^2]
or [emu] = [10^{-3} Am^2]

$$M = \frac{m}{V}$$

Magnetisation (EN)
Aimantation (FR)
[A/m]

Volume [m^3]

So: do not confuse the two m's : « M » and « m »



(argh!)

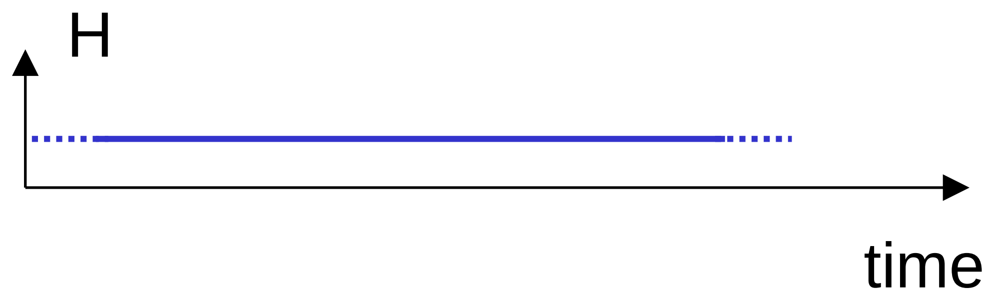
So: do not confuse the two m's : « M » and « m »

Beaucoup mieux:

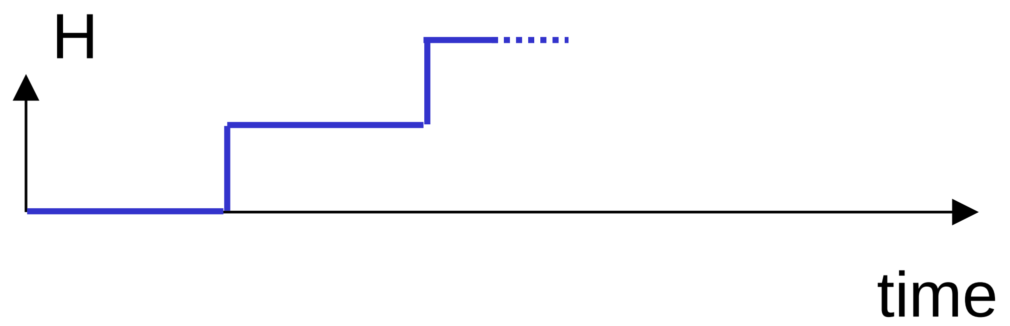
M&m's

Types of magnetic sollicitations

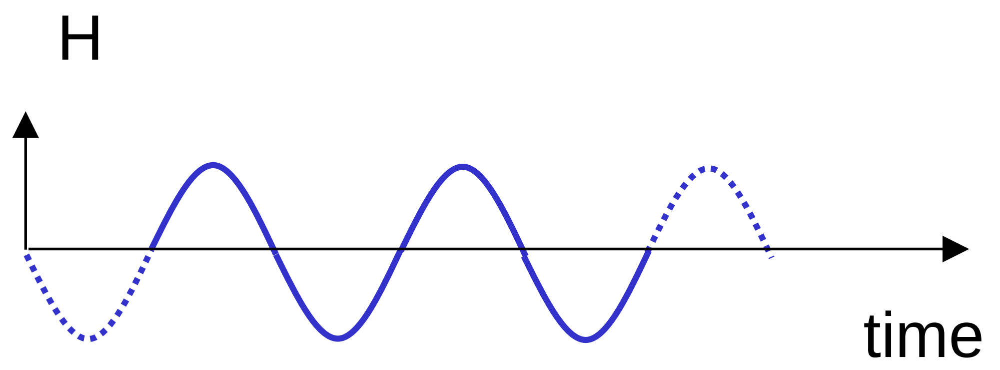
Direct current
Steady-state regime
→ « DC »



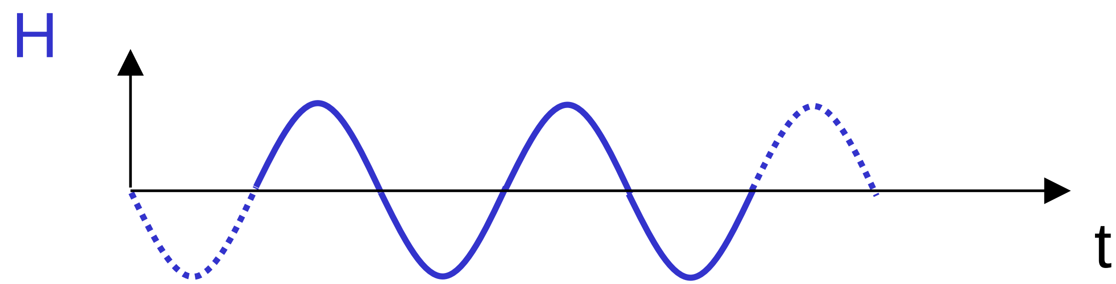
Quasi-static
Transient regime
→ « DC » (too)



Alternating
Sinewave signal
→ « AC »



Characteristics of AC susceptibility

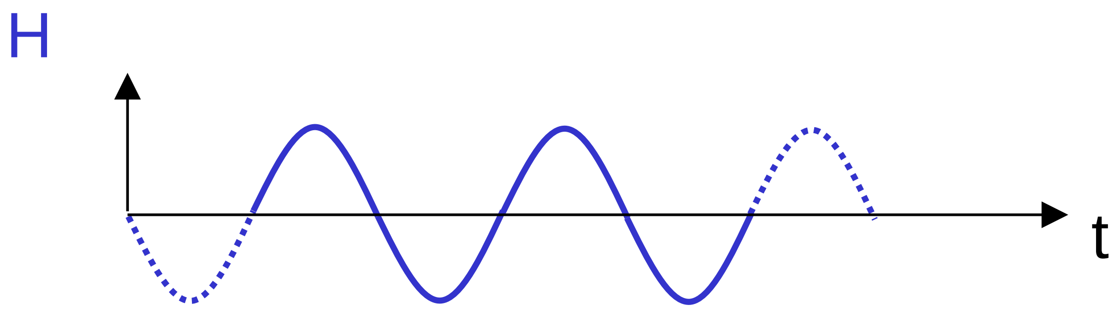


dt ➡ dH ➡ dM ➡ dB

sensitivity to (dM/dH)

sensitivity to phenomena related to (dB/dt)

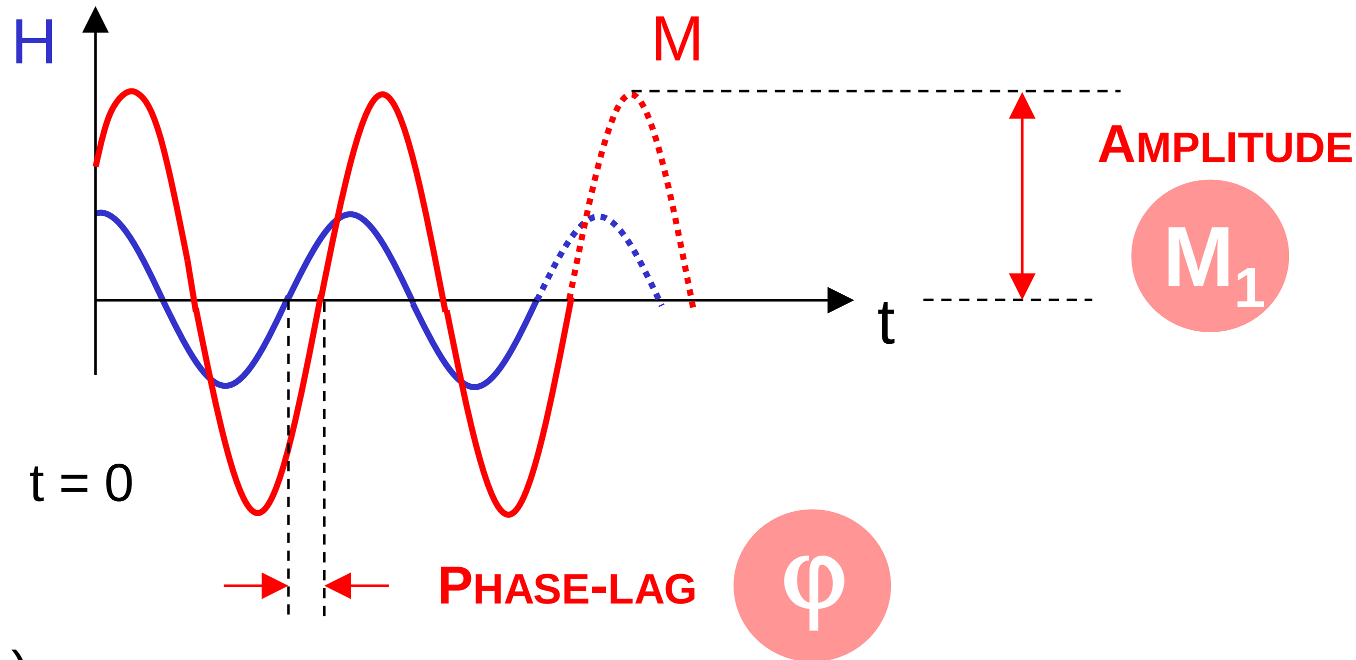
What are we measuring ? (1/2)



What are we measuring ? (1/2)

$H(t) = H_1 \cos(\omega t)$

[$\omega = 2\pi f$]



$M(t) = M_1 \cos(\omega t - \varphi)$

$= \underbrace{M_1 \cos(\varphi)}_{M'} \cos(\omega t) + \underbrace{M_1 \sin(\varphi)}_{M''} \sin(\omega t)$

! Important remarks !

- M(t) is assumed to follow a sinewave
- Physics dictates that M'' should be ≥ 0

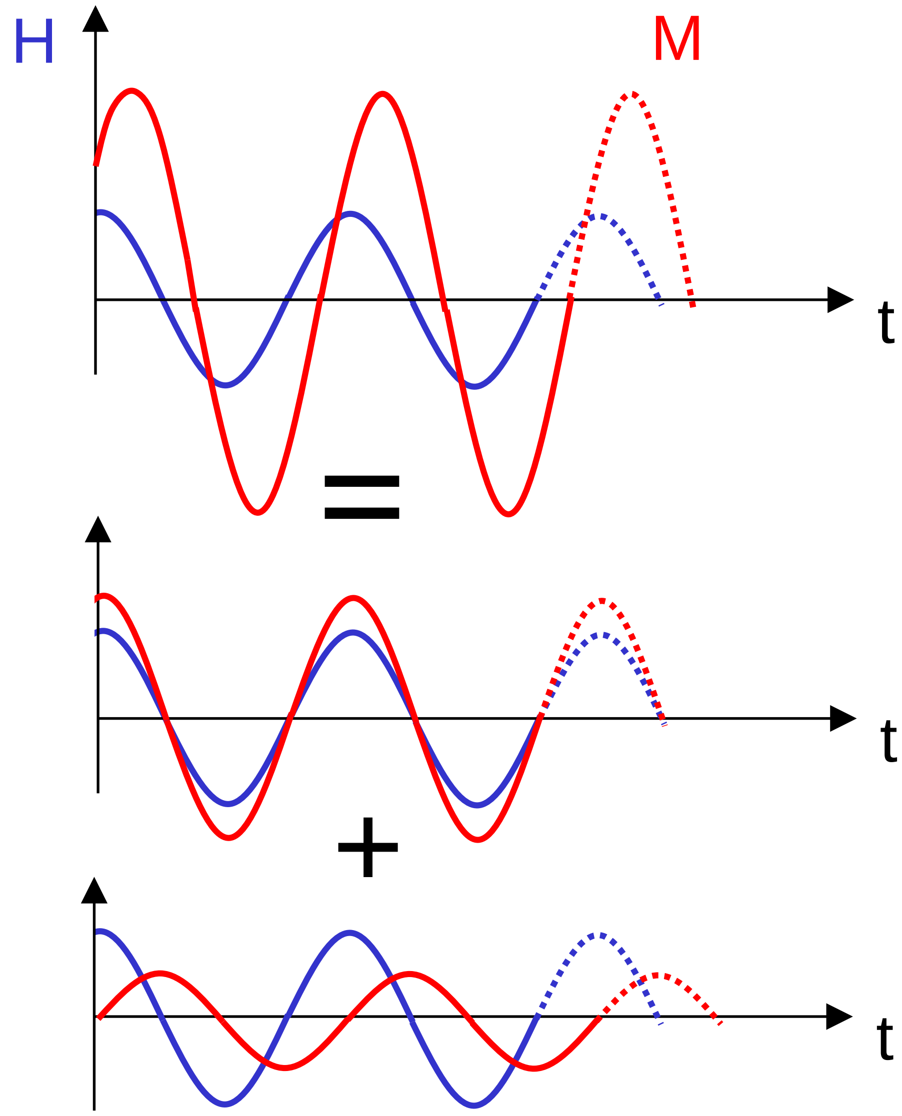
What are we measuring ? (1/2)

$H(t) = H_1 \cos(\omega t)$

$[\ \omega = 2\pi f\ ]$

$M' \cos(\omega t)$

$M'' \sin(\omega t)$



In-phase component

Out-of-phase component

Use of the complex notation (phasors)

Time notation		*Phasor notation*
$H(t) = H_1 \cos(\omega t) = \Re\,[\,e^{j\omega t}\,]$	⇒	$\overline{H_1} = H_1\,[\,e^{j\omega t}\,]$

Real number

$$M(t) = M_1 \cos(\omega t - \varphi)$$
$$= M_1 \cos(\varphi)\cos(\omega t) + M_1 \sin(\varphi)\sin(\omega t)$$
$$= M' \cos(\omega t) + M'' \sin(\omega t)$$

$$= \Re\,[\,M' e^{j\omega t}\,] + \Re\,[\,M'' e^{j(\omega t-\pi/2)}\,]$$
$$= \Re\,[\,M' e^{j\omega t}\,] + \Re\,[\,-j\,M'' e^{j\omega t}\,]$$
$$= \Re\,[\,(M' - j\,M'')\,e^{j\omega t}\,]$$

⇒ $\overline{M_1} = M_1\,[\,e^{j\omega t}\,]$

Complex number

$M_1 = M' - j\,M''$

$\chi_1 = M_1/H_1 = \chi' - j\,\chi''$

Complex AC susceptibility

When $M(t)$ is assumed to follow a pure sinewave
(i.e. only one « fundamental » signal at one frequency ω)
the complex AC susceptibility reads:

$$\chi_1 = \chi = \chi' - j\,\chi''$$

In-phase component (χ')

Out-of-phase component (χ'')

χ' is related to magnetic energy **stored in the material**

χ'' is related to magnetic energy **converted into heat**

during one cycle of
the applied AC field.

Signs of χ' and χ'' ?

The sign of χ' depends whether the material attracts or repels magnetic flux lines

χ' < 0 for diamagnetic mat.

χ' > 0 for paramagnetic mat.
ferromagnets
anti-ferromagnets
ferrimagnets
etc.

If there are no losses:
χ'' = 0

If there are magnetic losses:
χ'' > 0

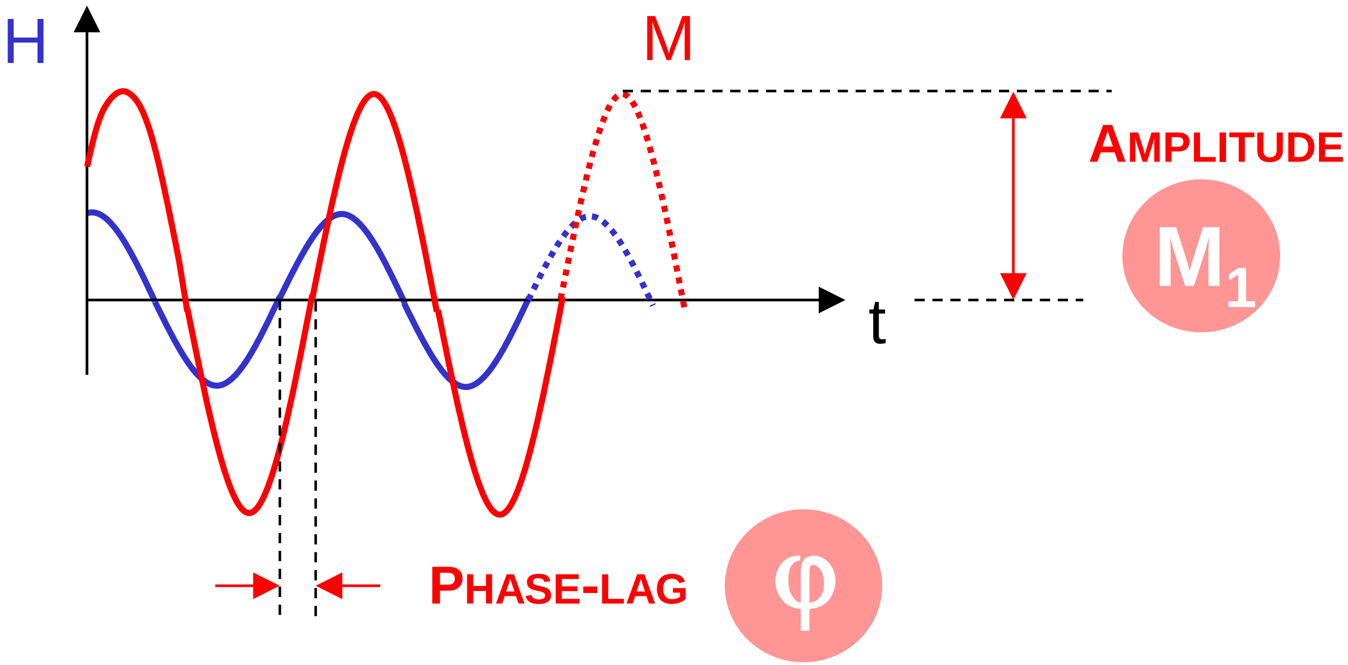
Energy W_q converted into heat during one cycle:

$$W_q = \pi(\mu_0 H_1^2)\chi'' > 0$$

- In **<u>all</u>** cases, **χ''** should be always ≥ **0**

What are we measuring ? (2/2)

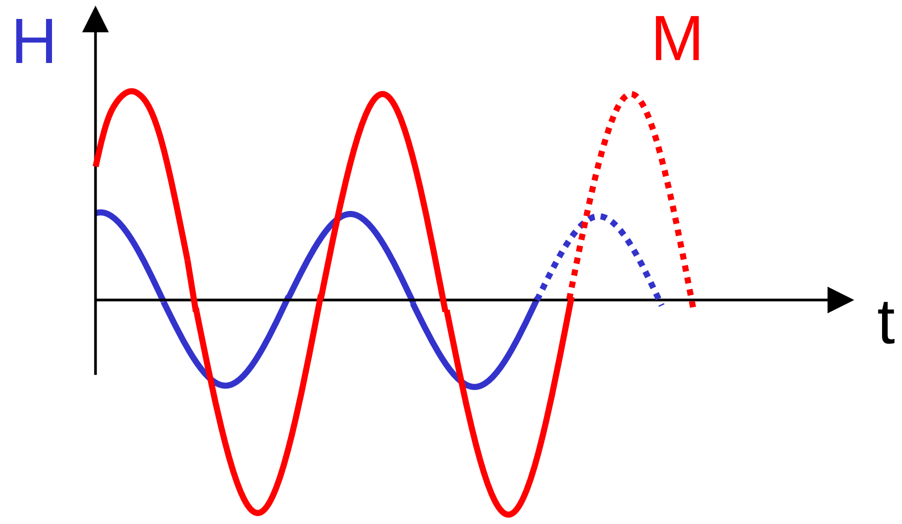
$H(t) = H_1 \cos(\omega t)$



- We will consider now that M(t) does **<u>NOT</u>** necessarily follow a sinewave

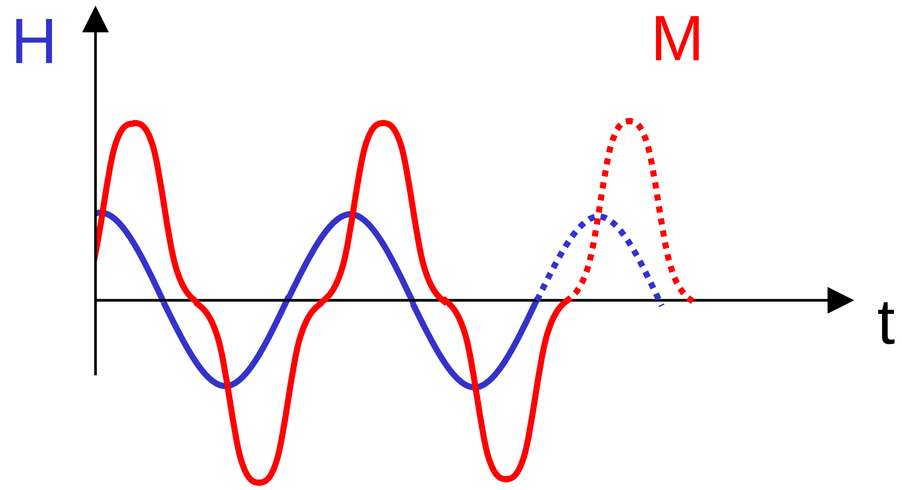
What are we measuring ? (2/2)

$H(t) = H_1 \cos(\omega t)$



What are we measuring ? (2/2)

$H(t) = H_1 \cos(\omega t)$



Since M(t) is not a sinewave, it can no longer be be expressed as
M' cos(ωt) + M'' sin(ωt)

BUT M(t) is still a periodic signal of the same period as the AC field.
Therefore, thanks to the Fourier theorem, M(t) reads

$$M(t) = H_1 \sum_{n=1}^{\infty} \left(\chi'_n \cos(n\omega t) + \chi''_n \sin(n\omega t) \right)$$

What are we measuring ? (2/2)

$H(t) = H_1 \cos(\omega t)$

$M(t)$

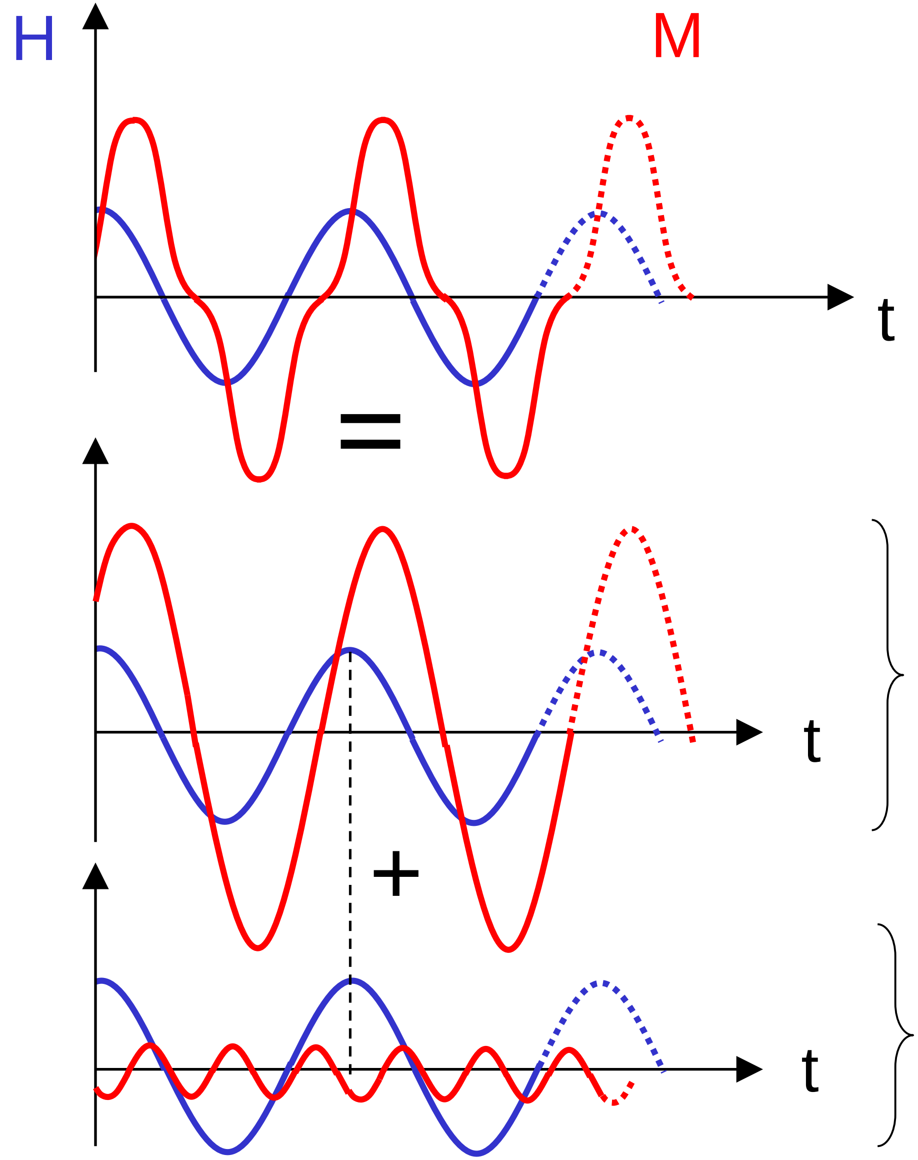
$=$

$M_1 \cos(\omega t - \varphi_1)$

Fundamental

$+ M_2 \cos(2\omega t - \varphi_2)$
$+ M_3 \cos(3\omega t - \varphi_3)$
$+ \ldots$

Harmonics



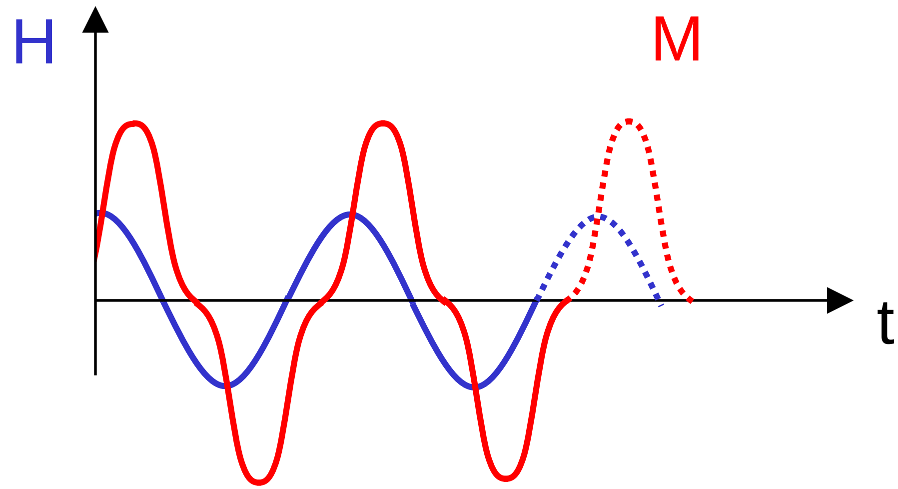
… with both in-phase and out-of-phase components

… with both in-phase and out-of-phase components

For the example displayed here, we assume that only the M_3 harmonics $\neq 0$

What are we measuring ? (2/2)

$H(t) = H_1 \cos(\omega t)$



$$M(t) = H_1 \sum_{n=1}^{\infty} \left(\chi'_n \cos(n\omega t) + \chi''_n \sin(n\omega t) \right)$$

If M(t) is a pure sinewave : χ'_1 and χ''_1 only $\rightarrow$ χ' (= χ'_1) and χ'' (= χ'_1)

If M(t) is distorted : $\rightarrow$ the harmonic susceptibilities might be $\neq 0$

- Harmonics originate from the **non-linearity** of the M-H process

How to find χ and χ'' in the general case ?

$$M(t) = H_1 \sum_{n=1}^{\infty} \left(\chi'_n \cos(n\omega t) + \chi''_n \sin(n\omega t) \right)$$

⇓

$$\chi'_1 = \frac{1}{\pi H_1} \int_0^{2\pi} M(\omega t)\ \cos(\omega t)\, d(\omega t)$$

$$\chi''_1 = \frac{1}{\pi H_1} \int_0^{2\pi} M(\omega t)\ \sin(\omega t)\, d(\omega t)$$

The knowledge of M(t) is resulting from H(t) is therefore required to predict theoretically the values of χ' and χ''.

How to find the harmonics components?

$$M(t) = H_1 \sum_{n=1}^{\infty} \left(\chi'_n \cos(n\omega t) + \chi''_n \sin(n\omega t) \right)$$

⇓

$$\chi'_n = \frac{1}{\pi H_1} \int_0^{2\pi} M(\omega t) \; \cos(n\omega t)\, d(\omega t)$$

$$\chi''_n = \frac{1}{\pi H_1} \int_0^{2\pi} M(\omega t) \; \sin(n\omega t)\, d(\omega t)$$

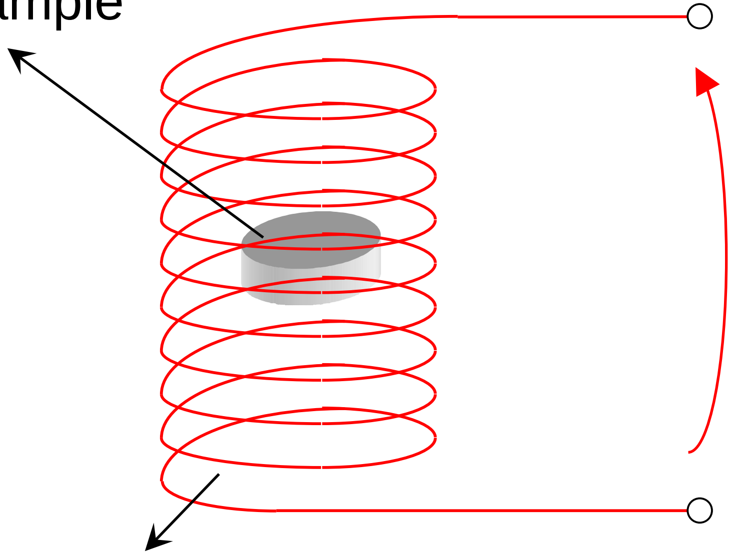
The analytical determination of the harmonics components will be carried out particular cases in the section « what kind of information ».

Outline

- ❑ What are we measuring?
- ❑ How are we measuring?
- ❑ What kind of information can we extract?
- ❑ Beyond the classic setup : variants and particular designs

Use of Faraday law: e.m.f. = - N dφ/dt

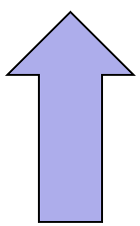
sample



pick-up coil
(N turns)

e.m.f.

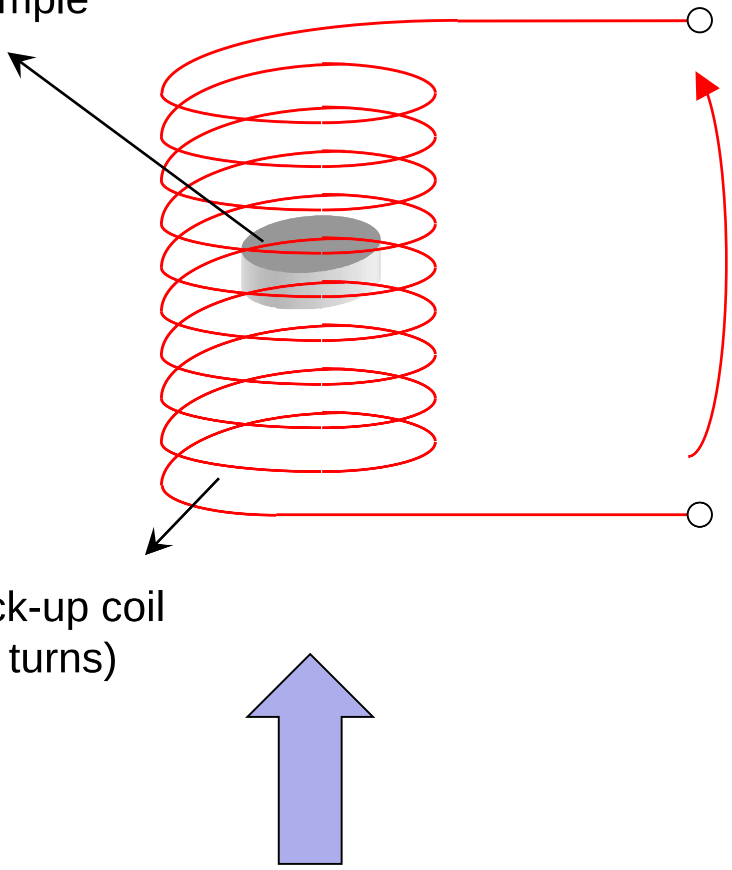
$$v(t) = \sum_{n=1}^{\infty} \left(v'_n \cos(n\omega t) + v''_n \sin(n\omega t) \right)$$



$H(t) = H_1 \cos(\omega t)$
produced by an air coil

- Fundamental (n=1) and harmonics components can be separated by filtering
- In-phase and out-of-phase components can be measured using a lock-in amplifier (PSD = Phase Sensitive Detection)

Use of Faraday law : e.m.f. = - N dϕ/dt



H(t) = H_1 cos(ωt)
produced by an air coil

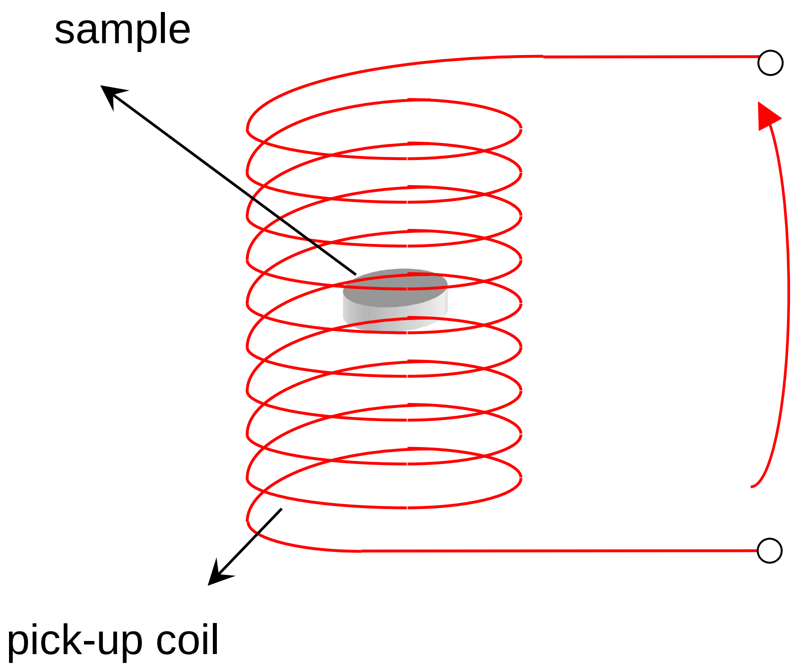
e.m.f.

$$v(t) = \sum_{n=1}^{\infty} \left(v'_n \cos(n\omega t) + v''_n \sin(n\omega t) \right)$$

Each component is a function of

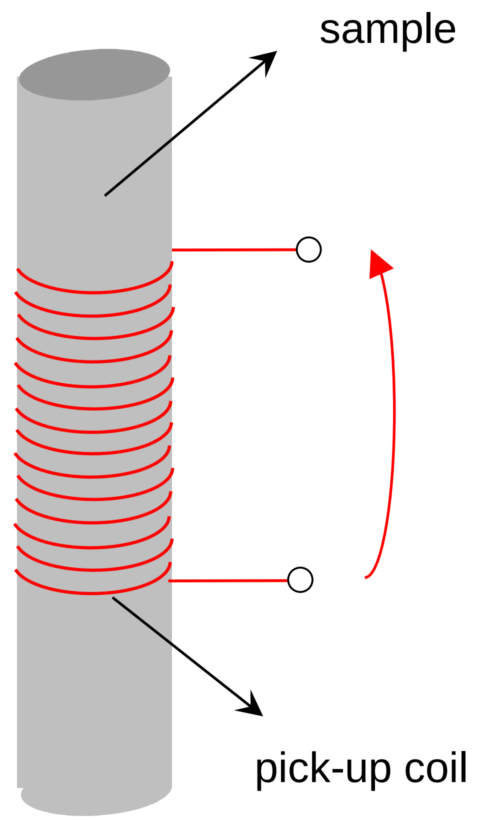
- field parameters : $\propto H_1$ and $\propto \omega$
- the sample volume $\propto$ V
- the number of turns of the coil $\propto$ N
- the sample dimensions
- the coil dimensions
- the magnetic susceptibility

Sample and pick-up coil dimensions: Two limiting cases



sample dimensions << coil dimensions

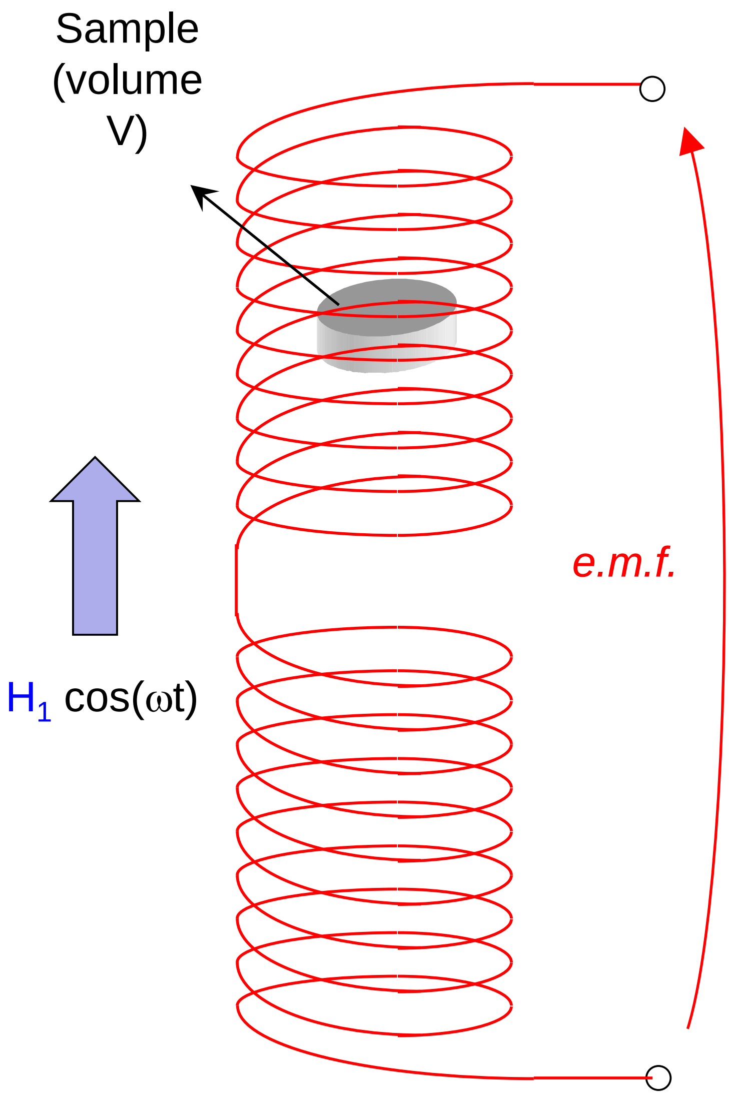
Sensitive to the magnetic moment m ∝ <M>



Tightly wrapped coil

Sensitive to the magnetic flux ϕ ∝ <B>

Configuration the most sensitive to χ



Two identical pick-up coils are placed in series opposition. One can show that the e.m.f. v is given by

$$v = \left(\frac{1}{\alpha}\right) V\, f\, H_1\, \chi$$

where α is the calibration constant of the susceptometer, which depends primarily of the geometry of the sensing coils. It has to be determined by an appropriate calibration procedure. Note that the calibration factor derived is valid strictly only for specimens of the same size and shape as the standard used.

Calibration of the susceptometer

Calibration of ac susceptometer for cylindrical specimens

R. B. Goldfarb and J. V. Minervini

Electromagnetic Technology Division, National Bureau of Standards, Boulder, Colorado 80303

(Received 11 November 1983; accepted for publication 30 January 1984)

The absolute magnetic susceptibility of cylindrical specimens is obtained with an ac susceptometer whose calibration is based on a calculation of mutual inductance. An axially magnetized cylinder is modeled as a solenoid of the same size. The mutual inductance between such a solenoid and a pickup coil of arbitrary dimensions is computed. The susceptibility is then a function of the mutual inductance, the cylinder length, the magnitude and frequency of the ac magnetizing field, and the voltage induced on the pickup coil. Demagnetization factor and eddy-current effects are considered, an example is given, and pickup coil compensation is discussed. Other calibration methods are also presented.

PACS numbers: 07.55. + x

Goldfarb R B and Minervini J V (1984) *Rev. Sci. Instrum.* 55 761

Calibration of the susceptometer

REVIEW OF SCIENTIFIC INSTRUMENTS **82**, 045112 (2011)

Calibration of ac and dc magnetometers with a Dy_2O_3 standard

http://dx.doi.org/1

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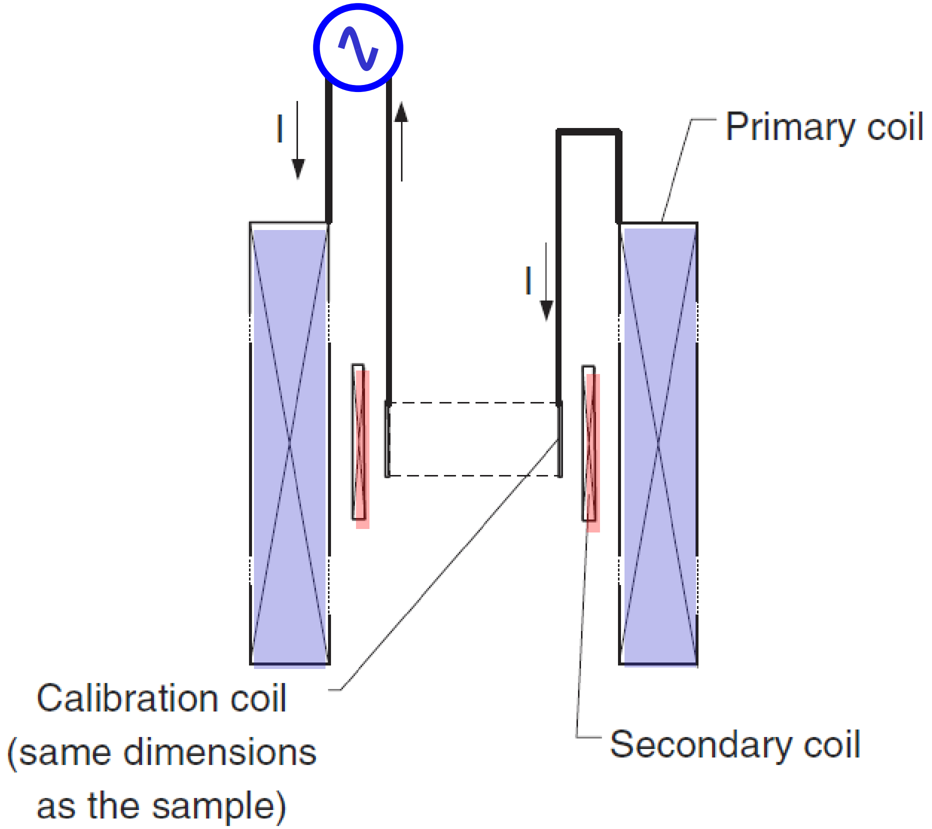
(Received 29 November 2010; accepted 27 March 2011; published online 27 April 2011)

The ac susceptibility and magnetization curves of a glued Dy_2O_3 powder sample are measured by an ac susceptometer and a dc superconducting quantum interference device magnetometer, both of which have been calibrated previously. It is shown that the magnetic moment of the paramagnetic sample as a function of field and temperature may be accurately expressed by a combination of the Curie–Weiss law and the Langevin function at $T > 45$ K with three adjusting parameters, so that the dc magnetization curves and the magnitude and phase of ac susceptibility at different values of dc bias field measured by any magnetometer can be calibrated by using Dy_2O_3 as a standard. The

D.-X. Chen, V. Skumryev, and B. Bozzo, *Rev. Sci. Instrum.* **82** 045112 (2011)

Calibration of the susceptometer

- <u>Idea</u> : using a calibration coil connected **electrically in series** with the primary coil of the susceptometer. This ensures that the calibration ac magnetic moment *m* (produced by the calibration coil) has exactly the same frequency and the same phase as the applied AC magnetic field.



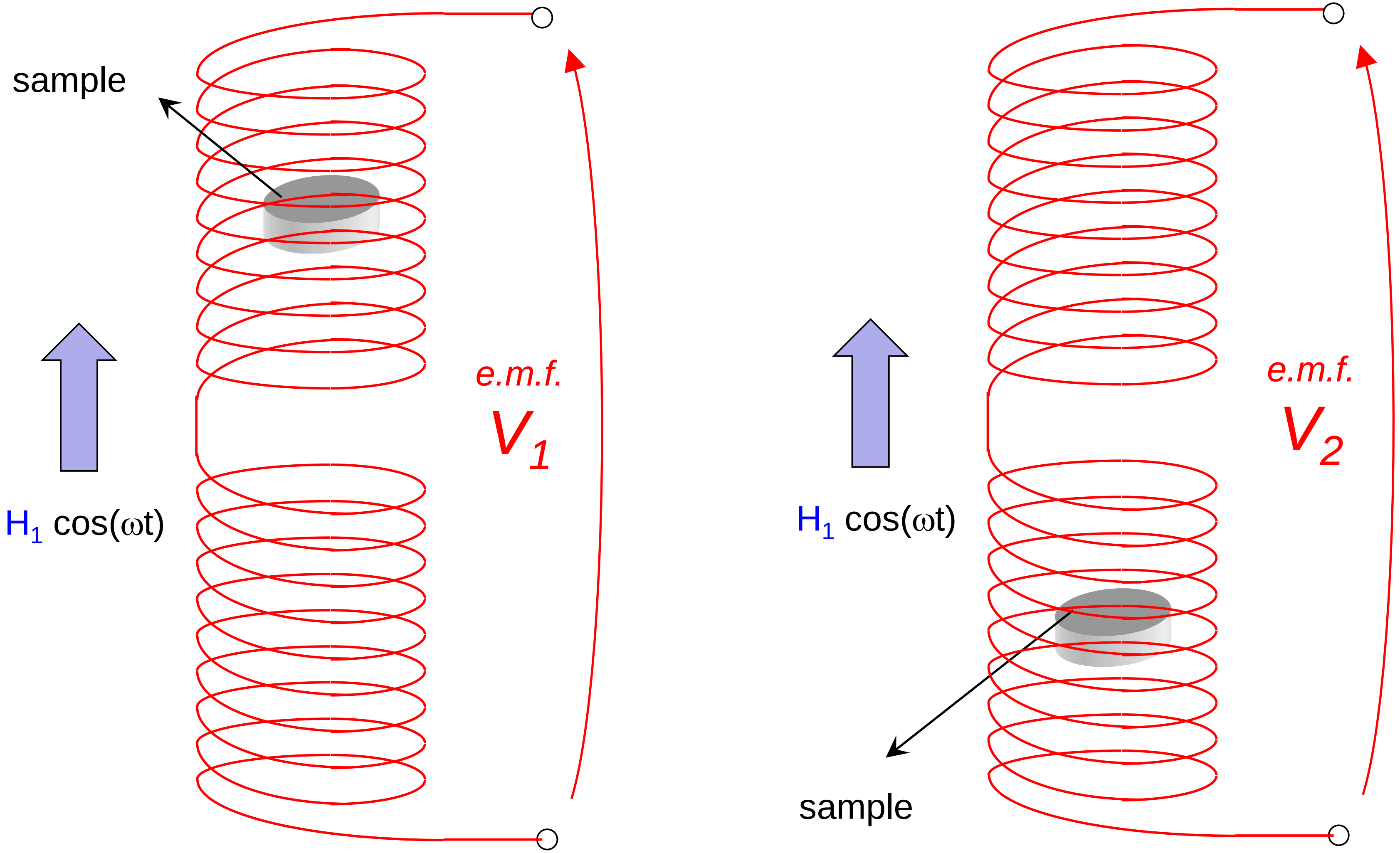
If *I* represents the current in the calibration coil of cross section *S*, and N turns, the resulting magnetic moment, m_{cal}, is then given by

$$m_{cal} = NIS.$$

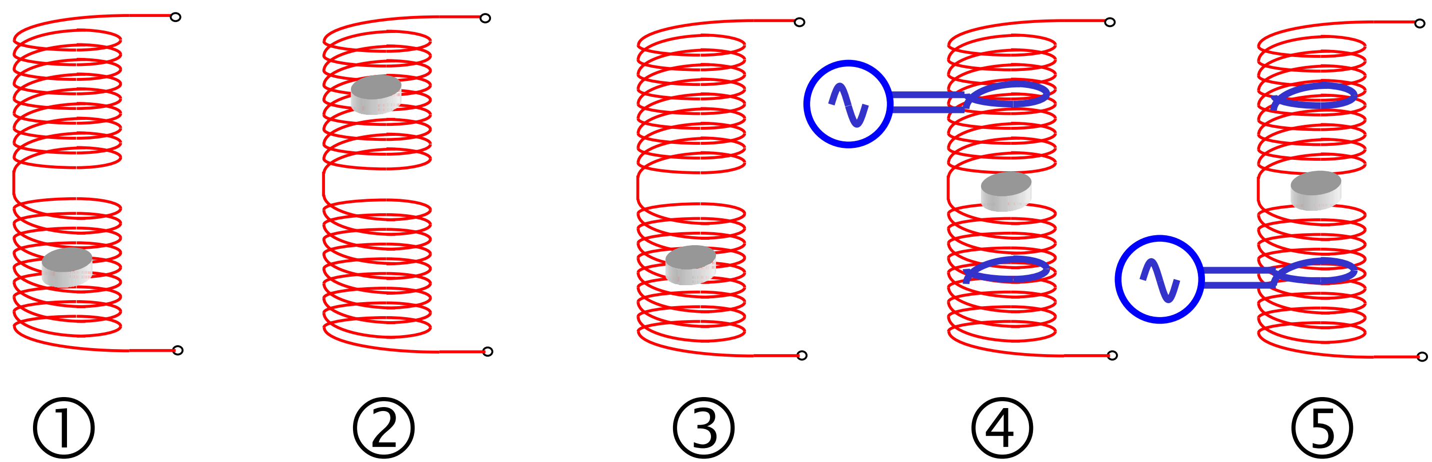
P Laurent, J F Fagnard, B Vanderheyden, N Hari Babu, D A Cardwell, M Ausloos and P Vanderbemden, *Measurement Science and Technology* **19** (2008) 085705

Overcoming the unbalance of pick-up coils

Two **identical** pick-up coils can never be achieved. The best method is to place at the **centre of both sensing coils** and subtracting the signals : $V = (V_1 - V_2) / 2$.



Procedure carried out in a QD PPMS



By default, the sample undergoes a five-point measurement process that utilizes the calibration coil to increase measurement accuracy. The first reading is made with the sample positioned in the center of the bottom detection coil. Then the sample is positioned in the center of the top detection coil, and then in the center of the bottom coil again. During all three readings, the signals from the detection coil array are amplified, low-pass filtered, and digitized by an analog-to-digital converter (A/D). These signals are stored as waveform blocks in the data buffer. All 128 buffer points are used to record each response waveform. The points are fitted and compared to the driving signal to determine the real and imaginary components of the response when the sample is in the center of each detection coil. (Imaginary components are in phase with the driving signal and real components are 90° out of phase with the driving signal.) Subtracting one reading from the other gives a sample vector in the complex plane.

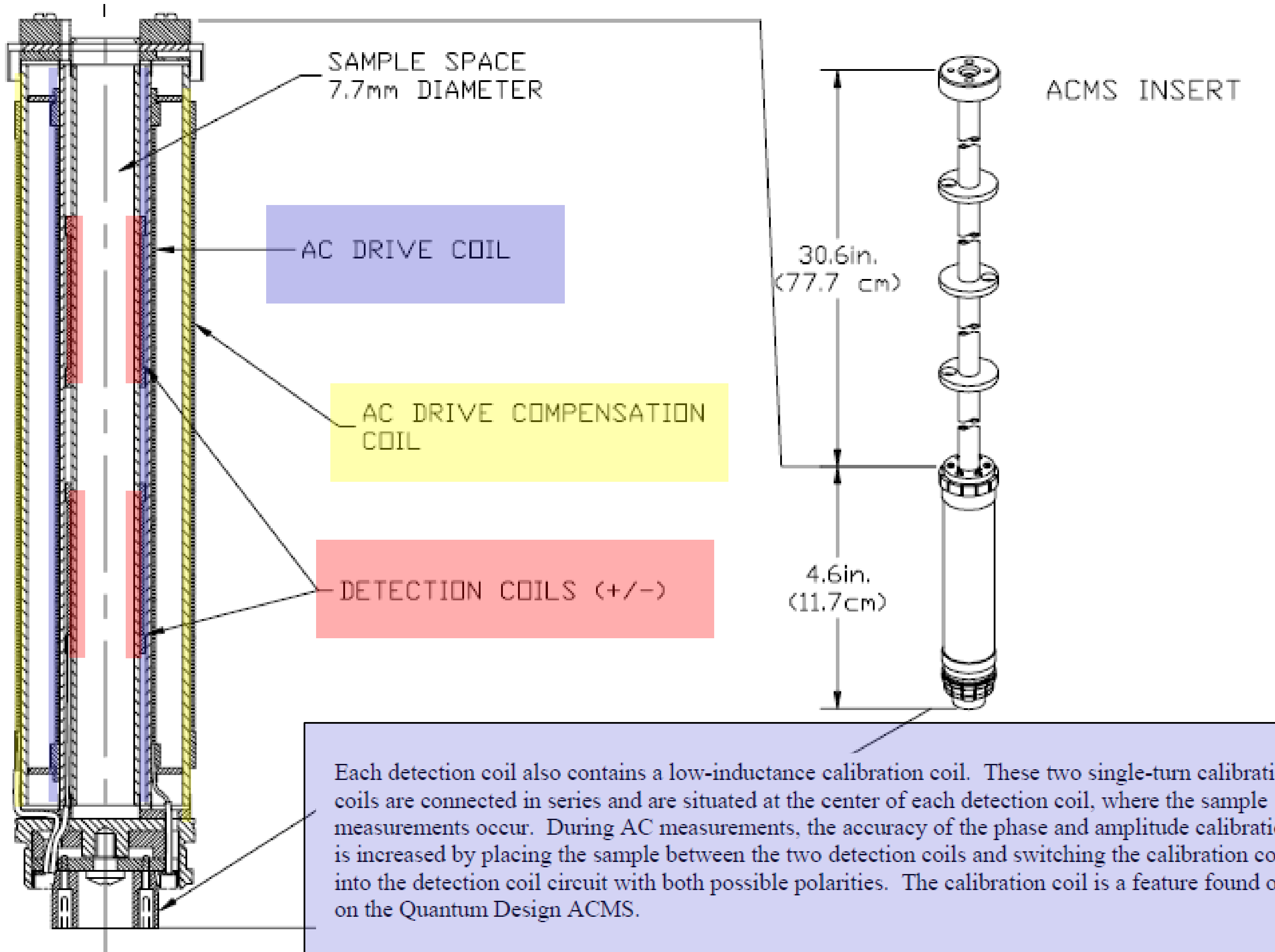
When the bottom-top-bottom coil readings are complete, the sample is placed at the center of the detection coil array so that it is between the two detection coils. Two more readings are taken with the calibration coil switched into the detection circuit with opposing polarities. The real and imaginary components of each response waveform are obtained by again fitting the data and comparing it to the driving signal. The two calibration readings are subtracted to yield a calibration vector in the complex plane. Subtracting the two calibration readings subtracts out the sample signal, leaving only environmental and instrumental factors that affect the reading.

① ② ③ } *Measurement*

④ ⑤ } *Calibration*

Physical Property Measurement System AC Measurement System Option User's Manual

In practice, 6 coils are used



Each detection coil also contains a low-inductance calibration coil. These two single-turn calibration coils are connected in series and are situated at the center of each detection coil, where the sample measurements occur. During AC measurements, the accuracy of the phase and amplitude calibration is increased by placing the sample between the two detection coils and switching the calibration coils into the detection coil circuit with both possible polarities. The calibration coil is a feature found only on the Quantum Design ACMS.

Figure 1-1. ACMS Insert and Coil Set

Physical Property Measurement System AC Measurement System Option User's Manual

Importance of centering the sample

- In a commercial device, the sample has to be centered by moving accurately through the detection coils, and record the signal for a finite number of well-defined (e.g. 64) positions.
- Note however that no notion is used for the measurement itself. In an AC susceptibility the sample is **stationary** during the measurement.
- That is why the "AC centering" is usually more accurate than the "DC centering"
- The centering has to be carried out each time a new sample is measured, ideally for the same **AC field amplitude** and **frequency** as for the measurement itself.

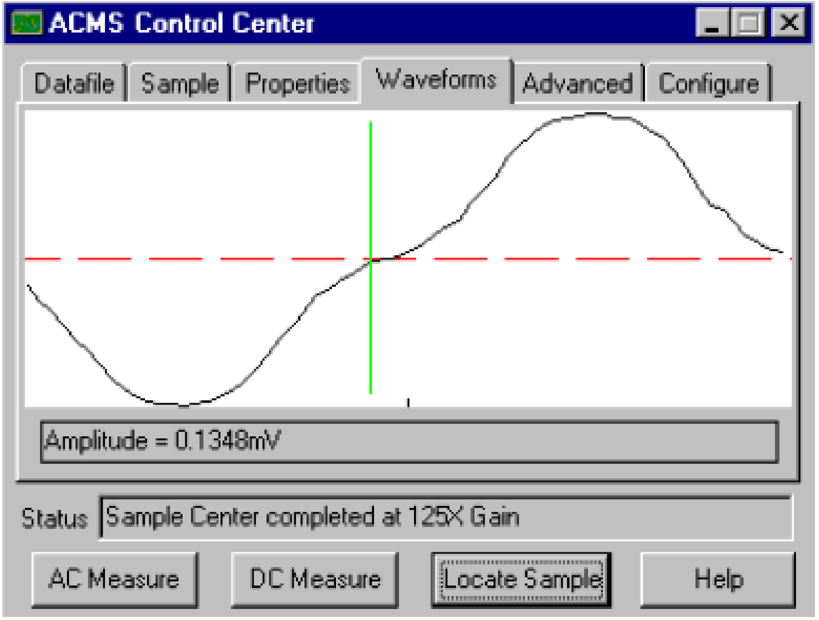


Figure 4-2. Example of Plot of AC Centering in **Waveforms** Tab

Outline

- What are we measuring?
- How are we measuring?
- What kind of information can we extract?
- Beyond the classic setup : variants and particular designs

The father of magnetism:

William Gilbert

(1544-1603)

« De Magnete »   (1600)

<u>NB</u> : Before Gilbert…

(about naturally magnetized stones)

« *This kind of stone restores husbands to wives and increases elegance and charm in speech.*

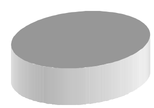
***Moreover, along with honey, it cures dropsy, fox mange and burn.* »**

Bartholomew of England (1203 – 1272)

What are the factors affecting the “magnetic response” to an AC field ?

$H(t) = H_1 \cos(\omega t)$

I am the sample



AC field amplitude

AC field frequency

DC field ?

sample properties

sample volume

sample geometry

Orientation of the field with respect to the sample

What are the factors affecting the "magnetic response" to an AC field ?

- Looking at the measured signal v across the sensing coils, one has

$$v = \left(\frac{1}{\alpha}\right) f \; V \; H_1 \; \chi$$

m M

m ………… = f (physics, applied field, volume)

M = m / V = f (physics, applied field, volume)

χ = M / H_1 = f (physics, applied field, volume)

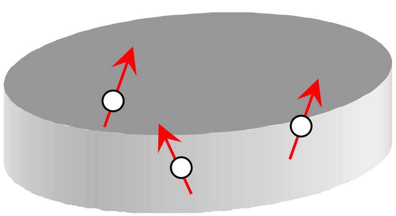
- In general, χ_{AC} may still depend on the applied field and on the volume !

Why does the AC susceptibility depend on the volume of the sample ?

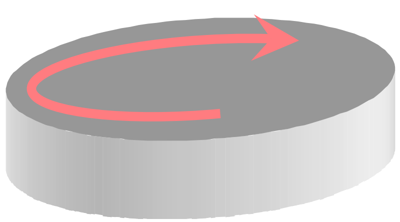
- One of the main reason is the **scale of the physical mechanism** giving rise to the AC magnetic response

Microscopic scale	***Macroscopic scale***
Dia- / Para- magnetism Ferro- / Ferri- magnetism Magnetic nanoparticules	Eddy currents in conductors Type-I superconductors Type-II superconductors

Orientation of spins



Macroscopic (shielding) currents



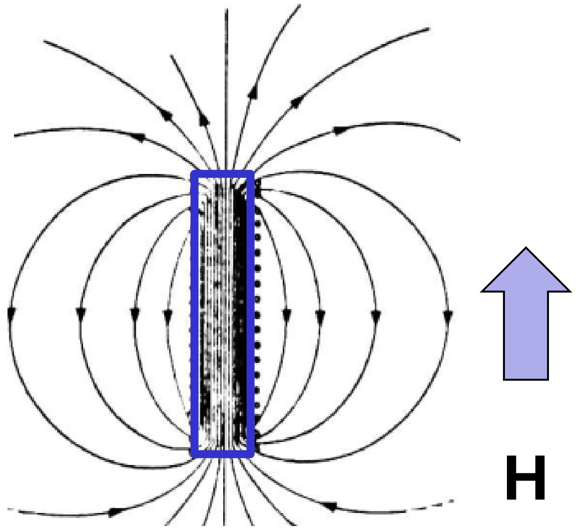
χ is **very weakly** volume dependent

χ is **strongly** volume dependent

Sample shape and field orientation: Demagnetization effects

A magnetized sample (e.g. M > 0) of **finite size** creates a field in the **surrounding space** and **within the sample** itself. This field – called **demagnetizing field** H_D – is always **opposite in direction to the sample magnetization**.

The **total** or **internal** applied field, H_T, is the sum of the field applied by the magnet H_{app}, **and** the demagnetizing field H_D. In the simple case H // H_D, one has



$$H_{int} = H_{app} + H_D$$

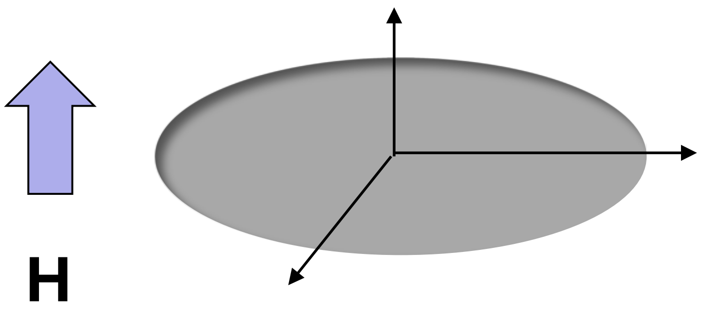
with

$$H_D = - D\ M$$

D represents the dimensionless **demagnetizing factor**

Demagnetizing factors “D” [or “N”]

Ellipsoid



- In this case M is uniform within the body. If M is parallel to one of the principal axes of the ellipsoid, the demagnetizing field is **uniform** too.
- The demagnetization factor D along the three axes can be calculated analytically.
- One has: $D_x + D_y + D_z = 1$.

PHYSICAL REVIEW VOLUME 67, NUMBERS 11 AND 12 JUNE 1 AND 15, 1945

Demagnetizing Factors of the General Ellipsoid

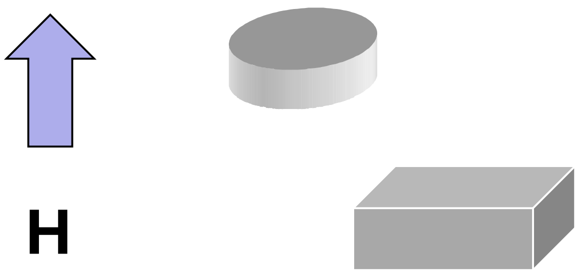
J. A. Osborn
Naval Ordnance Laboratory, Washington, D. C.
(Received March 24, 1945)

Charts and tables of the demagnetizing factors of prolate and oblate spheroids are readily available; however, demagnetizing factors of ellipsoids of three different axes are incompletely tabulated and laborious to calculate. This article presents charts and tables which make possible easy determination of the demagnetizing factor for any principal axis of an ellipsoid of any shape. Formulas for the demagnetizing factors of the general ellipsoid are included together with supplementary formulas which cover a large number of special cases.

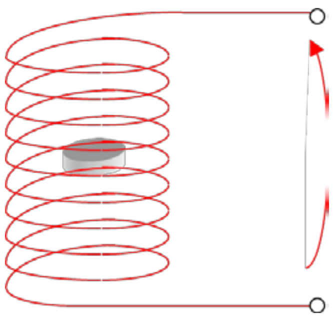
Osborn, J. A. Phys. Rev. **67**, 351-357, (1945)

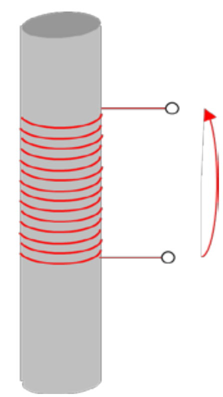
Demagnetizing factors “D” [or “N”]

Other shapes



- In general **M** is **NOT uniform** within the body.
- Therefore **averages of D** should be used.
- The simplest cases are still when the field is parallel to the principal axes of the body.
- One still has: $D_x + D_y + D_z = 1$.
- **Two kinds** of averages can be used, and therefore **two kinds** of demagnetization factors





MAGNETOMETRIC demagnetization factor

FLUXMETRIC demagnetization factor

Available online at www.sciencedirect.com
ScienceDirect
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Journal of Magnetism and Magnetic Materials 306 (2006) 135–146
Journal of magnetism and magnetic materials
www.elsevier.com/locate/jmmm

Fluxmetric and magnetometric demagnetizing factors for cylinders

D.-X. Chen[a,*], E. Pardo[b], A. Sanchez[b]

[a] ICREA and Grup d'Electromagnetisme, Departament de Física, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain
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Available online 23 March 2006

1. Introduction

As is described by Bozorth [1], when a rod is magnetized by an applied field H_a, its ends carry magnetic poles which themselves cause magnetic fields to be present in all parts of the rod. Normally, these fields are directed in the opposite direction to the field H_a, and are therefore called demagnetizing fields. Since demagnetizing fields are generally nonuniform, certain space averages are assumed for treating them. When the applied field H_a is uniform and the average is carried out over the midplane or the entire volume (with subscripts mid and vol), the demagnetizing field H_d is proportional to the magnetization M as

$$H_{\mathrm{mid,vol}} = H_a + H_{\mathrm{d,mid,vol}} = H_a - N_{\mathrm{f,m}} M_{\mathrm{mid,vol}}, \tag{1}$$

if the material has a constant susceptibility χ. Coefficients N_f and N_m are referred to as the fluxmetric and magnetometric demagnetizing factors, respectively, owing to their main applications in two types of magnetic measurements. We note that for samples with negative χ, H_d is in the direction along H_a but opposite to M. Thus,

Extremey useful references !

Chen D X, Brag J A and Goldfarb R B (1991) *IEEE Trans. Magn.* 27 3601
Chen D X, Pardo E and Sanchez A (2006) *J. Magn. Mater.* 306 135

JOURNAL OF APPLIED PHYSICS VOLUME 83, NUMBER 6 15 MARCH 1998

Demagnetizing factors for rectangular ferromagnetic prisms

Amikam Aharoni[a)]
Department of Electronics, Weizmann Institute of Science, 76100 Rehovoth, Israel

$$\pi D_z = \frac{b^2-c^2}{2bc}\ln\left(\frac{\sqrt{a^2+b^2+c^2}-a}{\sqrt{a^2+b^2+c^2}+a}\right) + \frac{a^2-c^2}{2ac}\ln\left(\frac{\sqrt{a^2+b^2+c^2}-b}{\sqrt{a^2+b^2+c^2}+b}\right) + \frac{b}{2c}\ln\left(\frac{\sqrt{a^2+b^2}+a}{\sqrt{a^2+b^2}-a}\right) + \frac{a}{2c}\ln\left(\frac{\sqrt{a^2+b^2}+b}{\sqrt{a^2+b^2}-b}\right)$$

$$+ \frac{c}{2a}\ln\left(\frac{\sqrt{b^2+c^2}-b}{\sqrt{b^2+c^2}+b}\right) + \frac{c}{2b}\ln\left(\frac{\sqrt{a^2+c^2}-a}{\sqrt{a^2+c^2}+a}\right) + 2\arctan\left(\frac{ab}{c\sqrt{a^2+b^2+c^2}}\right) + \frac{a^3+b^3-2c^3}{3abc}$$

$$+ \frac{a^2+b^2-2c^2}{3abc}\sqrt{a^2+b^2+c^2} + \frac{c}{ab}\left(\sqrt{a^2+c^2}+\sqrt{b^2+c^2}\right) - \frac{(a^2+b^2)^{3/2}+(b^2+c^2)^{3/2}+(c^2+a^2)^{3/2}}{3abc}.$$

Extremey useful too !

Journals & Magazines > IEEE Transactions on Magnetics > Volume: 38 Issue: 4

Demagnetizing factors of rectangular prisms and ellipsoids

3 Author(s) Du-Xing Chen ; E. Pardo ; A. Sanchez View All Authors

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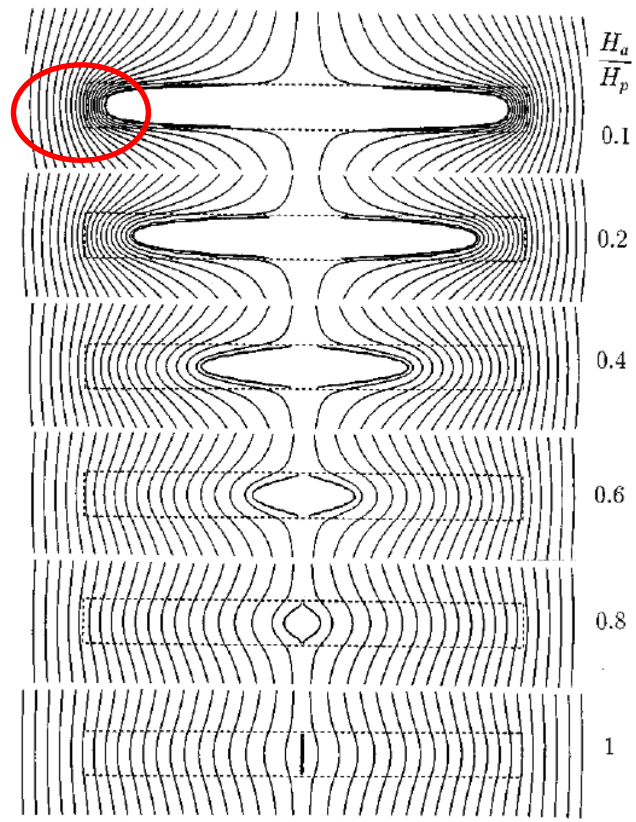
A. Aharoni Journal of Applied Physics 83, 3432 (1998)
D.-X. Chen, E. Pardo, and A. Sanchez, *IEEE Trans. Magn.*, 41, (2005) 2077

Since $H_{int} = H_{app} + H_D = H_{app} - D\,M$…

For ferromagnetic materials ($M > 0$),
H_{int} is <u>smaller</u> than H_{app} ("de-magnetizing")

while for superconductors in the diamagnetic state ($M < 0$),
H_{int} is <u>larger</u> than H_{app} ("re-magnetizing" ?).

Superconductor



<u>NB</u> : To understand magnetic flux penetration in Type-II superconductors of finite height, see…

PHYSICAL REVIEW B VOLUME 40, NUMBER 13 1 NOVEMBER 1989

Critical state in disk-shaped superconductors

M. Däumling and D. C. Larbalestier*
Applied Superconductivity Center, University of Wisconsin-Madison, Madison, Wisconsin 53706
(Received 24 July 1989)

We have calculated the magnetic fields and currents occurring in a disk-shaped superconductor (radius ≫ thickness) in the critical state in a self-consistent way using finite-element analysis. We find that the field shielded (or trapped) in the center of the disk is roughly equal to $J_c d$, where d is the thickness of the disk. The shielding currents also create radial fields which are of order $J_c d/2$ on the disk surface. For low applied fields $H_{appl} < J_c d$ these self-field effects dominate,

PHYSICAL REVIEW B VOLUME 58, NUMBER 10 1 SEPTEMBER 1998-II

Superconductor disks and cylinders in an axial magnetic field. I. Flux penetration and magnetization curves

Ernst Helmut Brandt
Max-Planck-Institut für Metallforschung, D-70506 Stuttgart, Germany
(Received 14 November 1997)

…. as well as all Helmut Brandt's papers ☺

Note however the important distinction :

Demagnetizing effects should always be taken into account when the sample cannot be considered infinitely long

BUT…

the conventional « demagnetizing factor » approach, strictly speaking, is valid for linear materials.

For type-II superconductors, only (semi-) analytical calculations and numerical modelling are appropriate !

Relation between χ_{int} and χ_{ext}

- One should always distinguish the **external (or apparent) susceptibility** χ_{ext} (obtained though a measurement) from the **internal susceptibility** χ_{int}

$$\chi_{ext} = \frac{M}{H_{app}} \qquad \chi_{int} = \frac{M}{H_{int}}$$

- From the relation $H_{int} = H_{app} - D\,M$, one finds directly:

$$\chi_{ext} = \frac{\chi_{int}}{1 + D\,\chi_{int}} \qquad \chi_{int} = \frac{\chi_{ext}}{1 - D\,\chi_{ext}}$$

Relation between χ_{int} and χ_{ext} for AC susceptibility

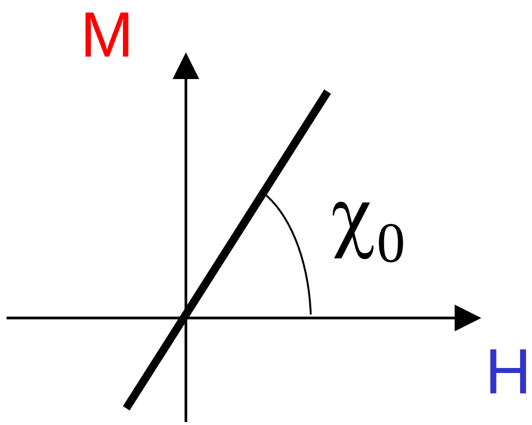
- In the AC regime, the values of external complex susceptibility $\chi_{ext} = \chi'_{ext} - j\,\chi''_{ext}$ can be converted into equivalent internal values $\chi_{int} = \chi'_{int} - j\,\chi''_{int}$ using the formulas

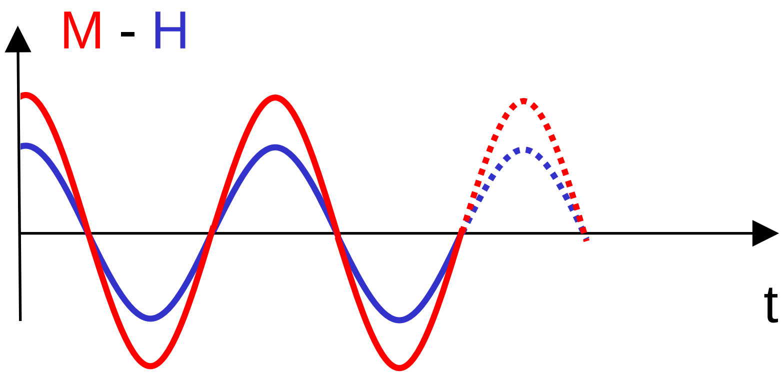
$$\chi'_{int} = \frac{\chi'_{ext} - D\left({\chi'_{ext}}^2 + {\chi''_{ext}}^2\right)}{D^2\left({\chi'_{ext}}^2 + {\chi''_{ext}}^2\right) - 2D\chi'_{ext} + 1}$$

$$\chi''_{int} = \frac{\chi''_{ext}}{D^2\left({\chi'_{ext}}^2 + {\chi''_{ext}}^2\right) - 2D\chi'_{ext} + 1}$$

A few examples of the theoretical response of different materials to an AC magnetic field

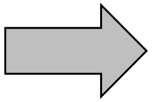
(1) a linear (ω independent) magnetic material





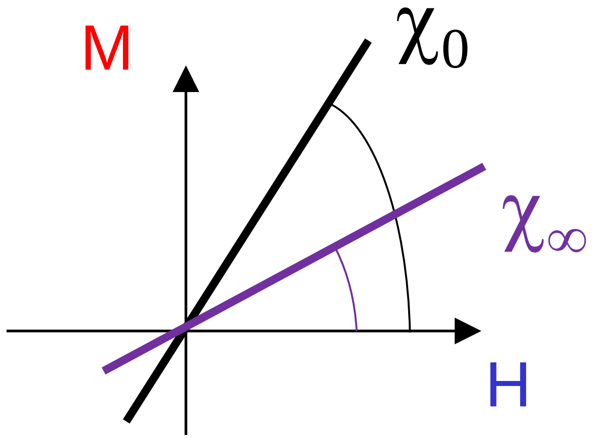
$\chi' = \chi_0$ and $\chi'' = 0$

$\chi'_n = 0$ and $\chi''_n = 0$



- No out-of-phase susceptibility
- No harmonics

(2) a linear, ω dependent, paramagnetic material



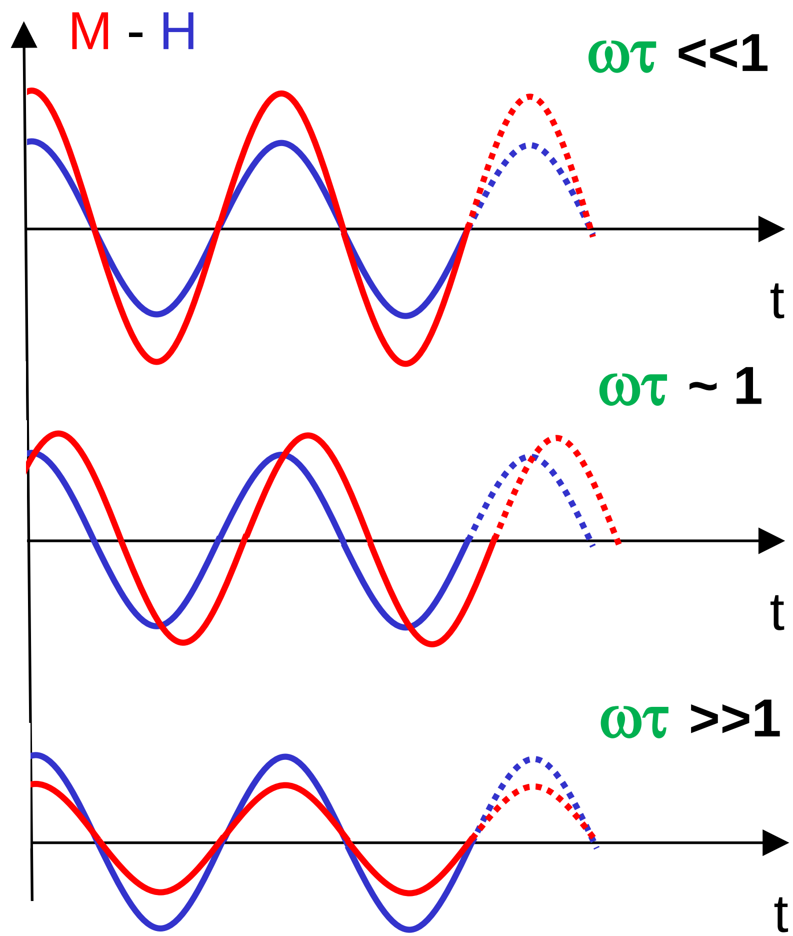
- We assume a Debye-type relaxation:

$$\chi = \chi_\infty + \frac{\chi_0 - \chi_\infty}{1 + j\omega\tau}$$

$$\chi' = \chi_\infty + \frac{\chi_0 - \chi_\infty}{1 + \omega^2\tau^2}$$

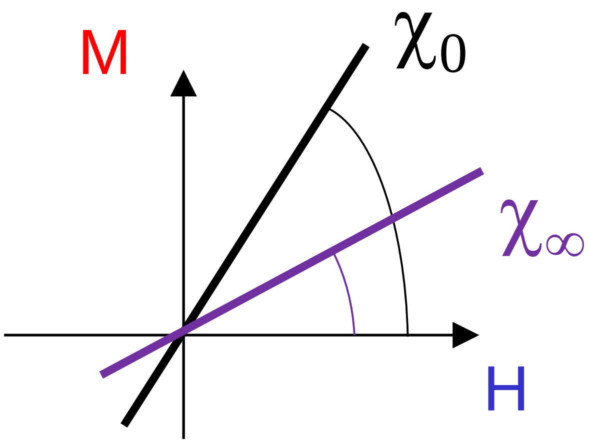
$$\chi'' = \frac{\omega\tau(\chi_0 - \chi_\infty)}{1 + \omega^2\tau^2}$$

$\chi'_n = 0$ and $\chi''_n = 0$



No harmonics

(2) a linear, ω dependent, paramagnetic material



$$\chi' = \chi_\infty + \frac{\chi_0 - \chi_\infty}{1 + \omega^2\tau^2}$$

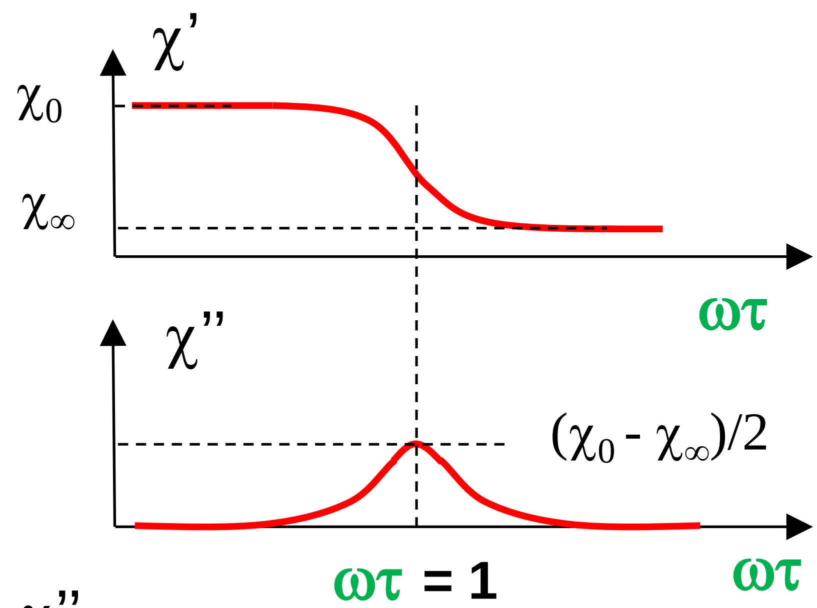
$$\chi'' = \frac{\omega\tau(\chi_0 - \chi_\infty)}{1 + \omega^2\tau^2}$$

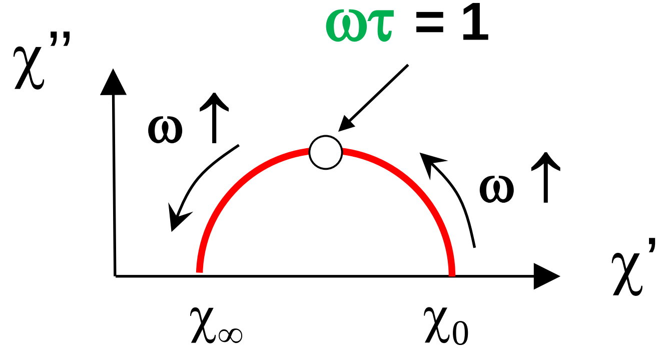
$\chi'_n = 0$ and $\chi''_n = 0$

➔ No harmonics

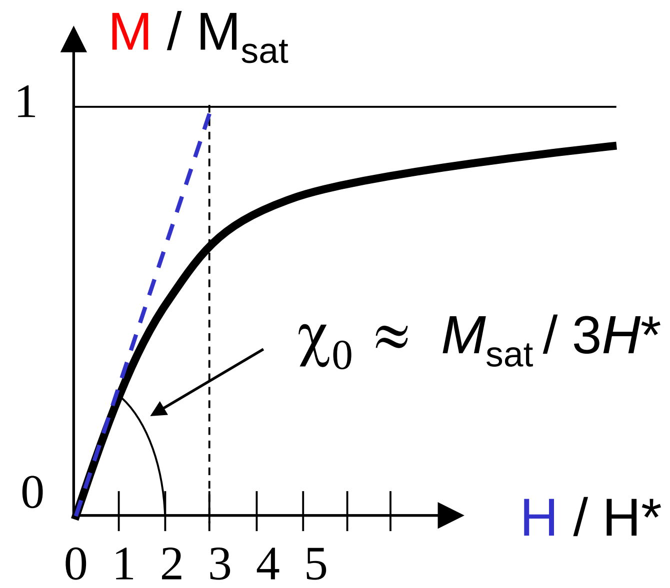
- We assume a Debye-type relaxation:

$$\chi = \chi_\infty + \frac{\chi_0 - \chi_\infty}{1 + j\omega\tau}$$





(3) a non-linear (ω independent) superparamgnetic mat.



$$M = M_{sat}\,\mathcal{L}\,(H / H^*)$$

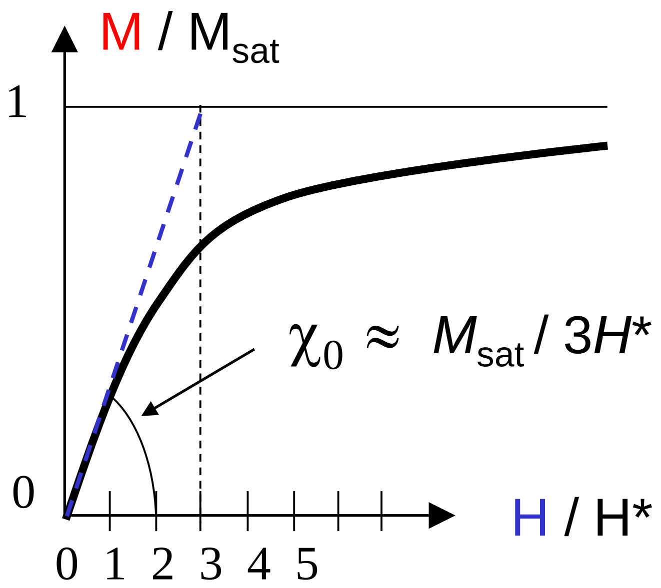
$$\mathcal{L}\,(x) = \coth(x) - 1/x$$
$$\approx (1/3)\,x - (1/45)\,x^3 + \ldots$$

$$M_{sat} = n\,m$$

$$H^* = k_B T / \mu_0 m$$

- n = density of magnetic moments
- m = individual magnetic moment
- k_B = Boltzmann constant
- T = absolute temperature
- $\mathcal{L}(x)$ = Langevin function

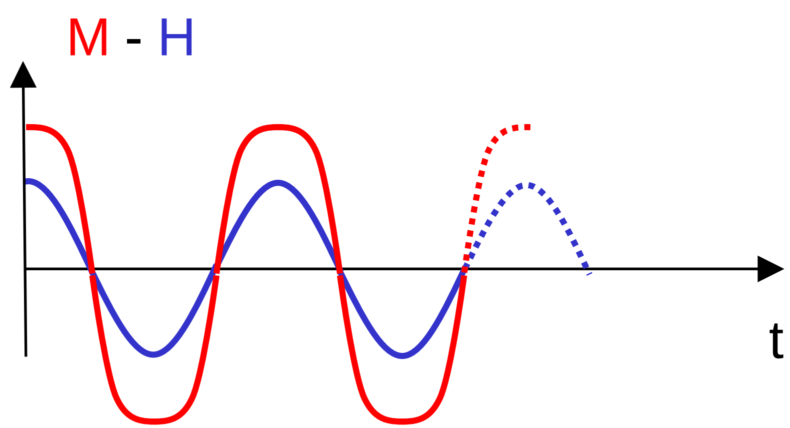
(3) a non-linear (ω independent) paramagnetic material



$M = M_{sat}\, \mathcal{L}\,(H / H^*)$

$\mathcal{L}\,(x) = \coth(x) - 1/x$

$\approx (1/3)\, x - (1/45)\, x^3\ [+\ldots]$



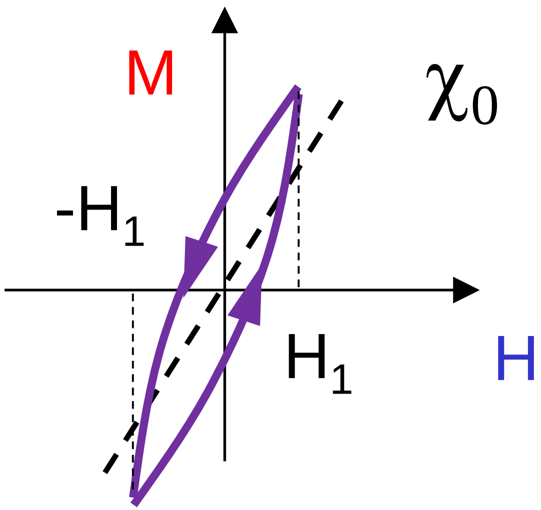
- Replacing H by $H_1 \cos(\omega t)$, and remembering that $\cos^3(x) = (3/4)\cos(x) + (1/4)\cos(3x)$, only the $\cos(\omega t)$ and the $\cos(3\omega t)$ terms remain. Thus

$$\chi' = \frac{M_{sat}}{3H*}\left[1 - \frac{{H_1}^2}{20H^{*2}}\right] \quad , \chi'' = 0$$

$$\chi_3' = -\frac{M_{sat}}{180\, H^{*2}}{H_1}^2 \quad , \chi_3'' = 0$$

- Harmonic 3
- No out-of-phase signal

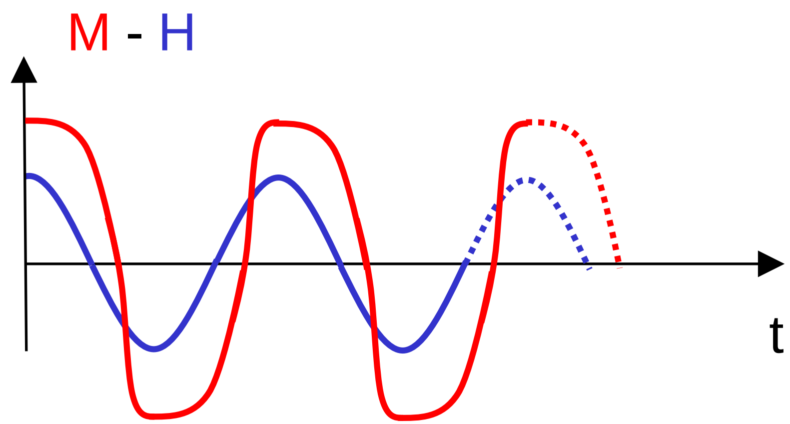
(4) a soft (insulating) ferromagnetic material



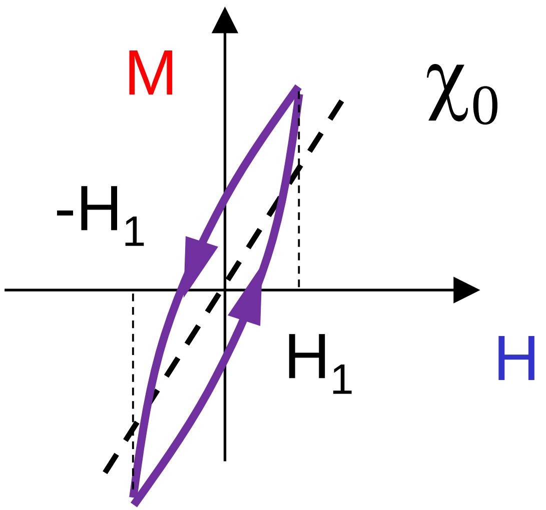
- We assume small amplitude hysteresis loops which are a quadratic function of H (**Rayleigh loops**)

$$M_{\downarrow} = \left(\chi_0 + \frac{H_1}{H^*}\right) H - \frac{1}{2H^*}\left(H^2 - H_1^2\right)$$

$$M_{\uparrow} = \left(\chi_0 + \frac{H_1}{H^*}\right) H + \frac{1}{2H^*}\left(H^2 - H_1^2\right)$$



(4) a soft (insulating) ferromagnetic material



- We assume small amplitude hysteresis loops which are a quadratic function of H (**Rayleigh loops**)

$$M_{\downarrow} = \left(\chi_0 + \frac{H_1}{H^*}\right) H - \frac{1}{2H^*}\left(H^2 - H_1^2\right)$$

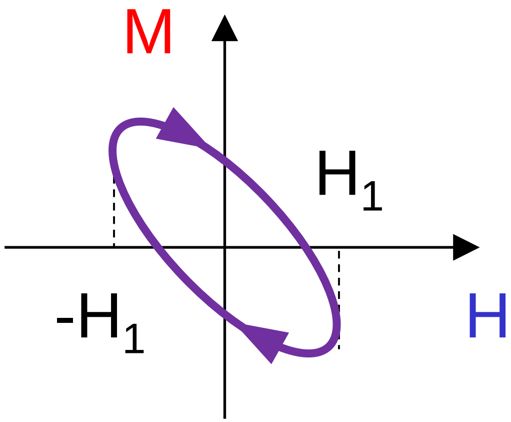
$$M_{\uparrow} = \left(\chi_0 + \frac{H_1}{H^*}\right) H + \frac{1}{2H^*}\left(H^2 - H_1^2\right)$$

$$\chi' = \chi_0 + \frac{H_1}{H^*} \qquad \chi'_3 = 0$$

$$\chi'' = \frac{4}{3\pi}\left(\frac{H_1}{H^*}\right) \qquad \chi''_3 = \frac{4}{15\pi}\left(\frac{H_1}{H^*}\right)$$

- Non-zero odd harmonics (3, 5, 7 etc.)
- Non-zero out-of-phase signal → losses!
- NB If $H_{DC} = 0$ → half-wave symmetry M(t-T/2) = - M(t)
  → No even harmonic

(5) a non-magnetic conducting material



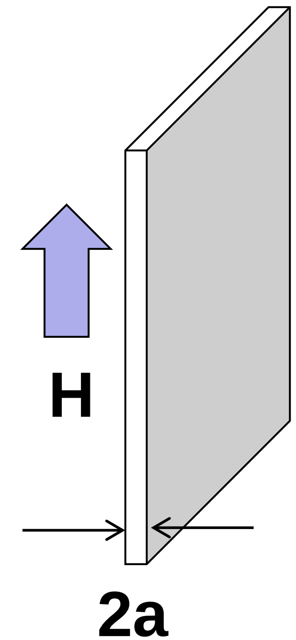
$H(t) = H_1 \cos(\omega t)$

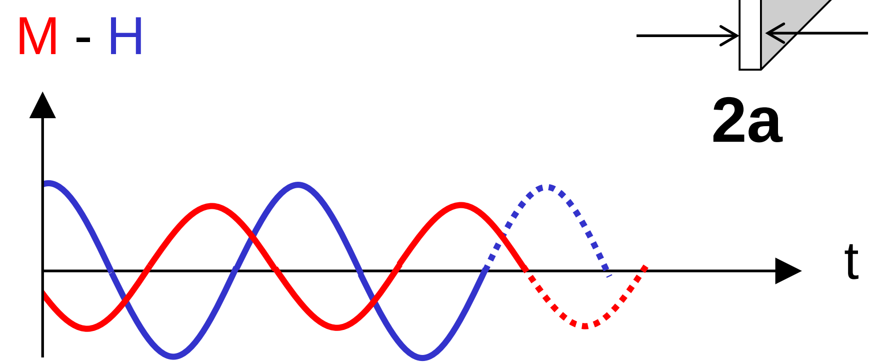
→ Eddy currents

→ Magnetization opposing to the variations of the applied field

→ Sample size and shape dependent

→ Here, we consider an infinite slab || H





$$\langle\chi\rangle = \frac{1}{ka}\tanh(ka) - 1$$

$$k = \frac{1+j}{\delta} \qquad \delta = \sqrt{\frac{2}{\omega\mu\sigma}}$$

- δ is the skin depth
- At low frequency , i.e. (a << δ), (ka << 1)

$$\langle\chi\rangle \approx -\frac{1}{3}(ka)^2 \approx -j\,\frac{2}{3}\left(\frac{a}{\delta}\right)^2$$

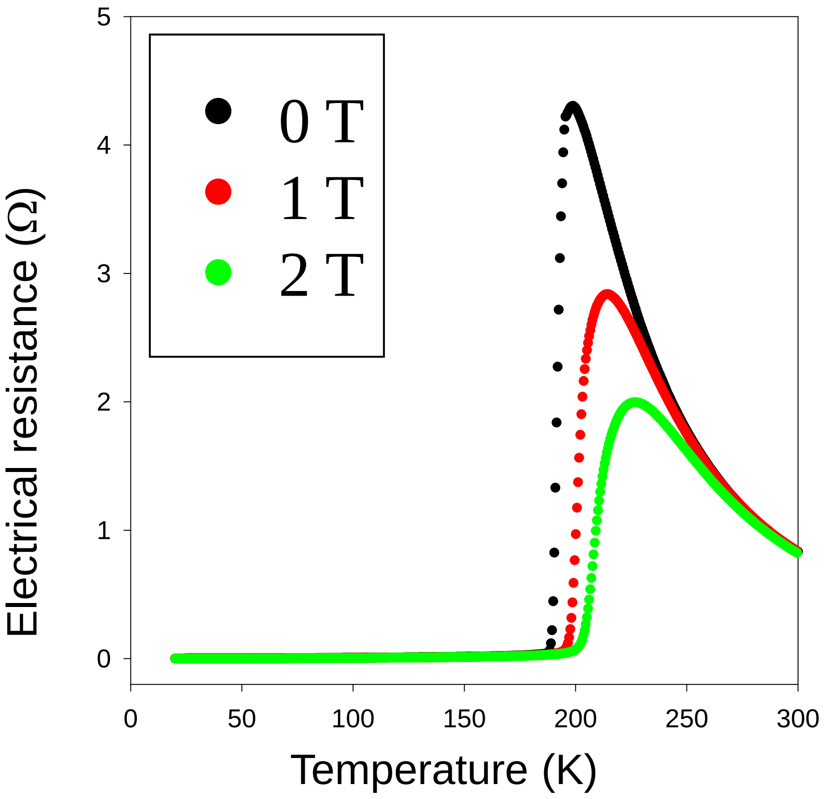
No harmonics

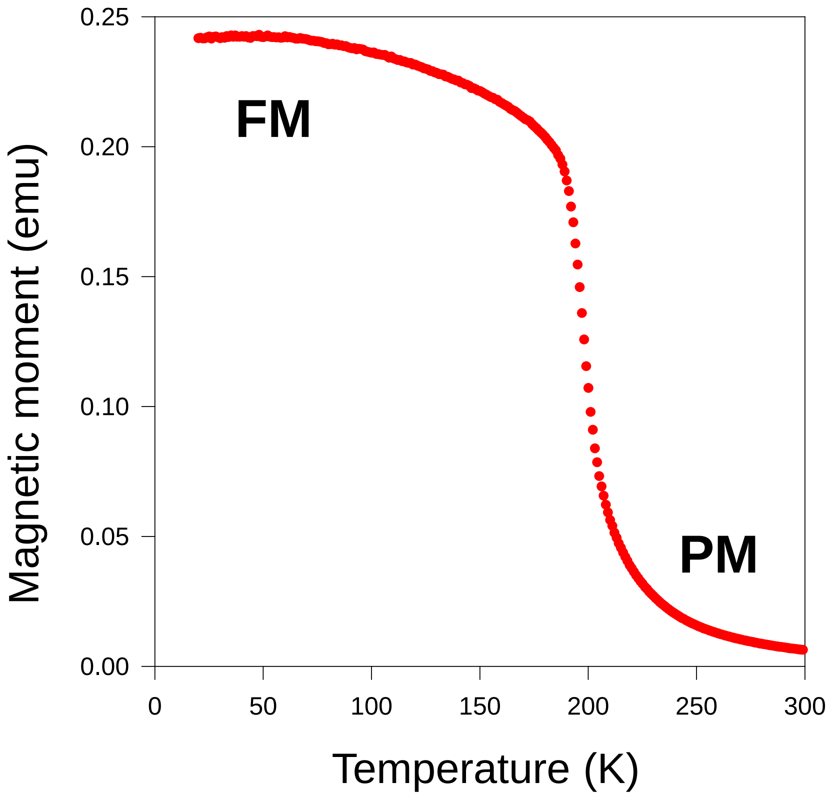
Non-zero out-of-phase signal

An example of the advantage of applying a DC field to an AC magnetic field

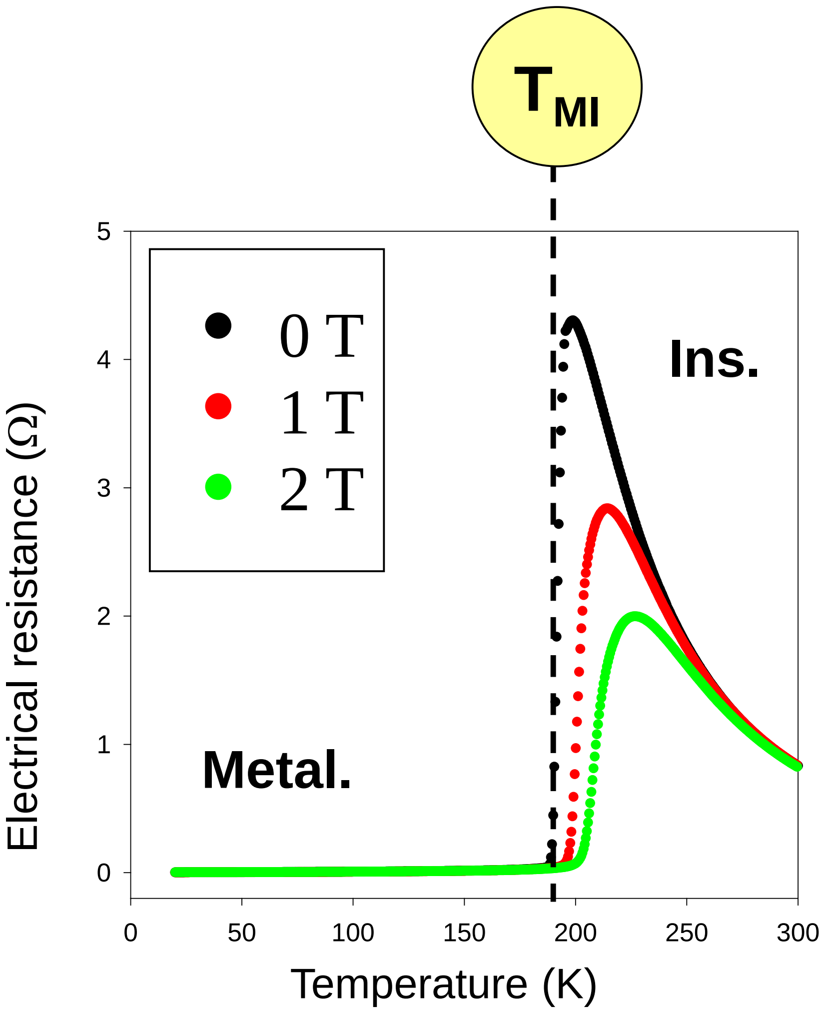
La-Ca-Mn-O system

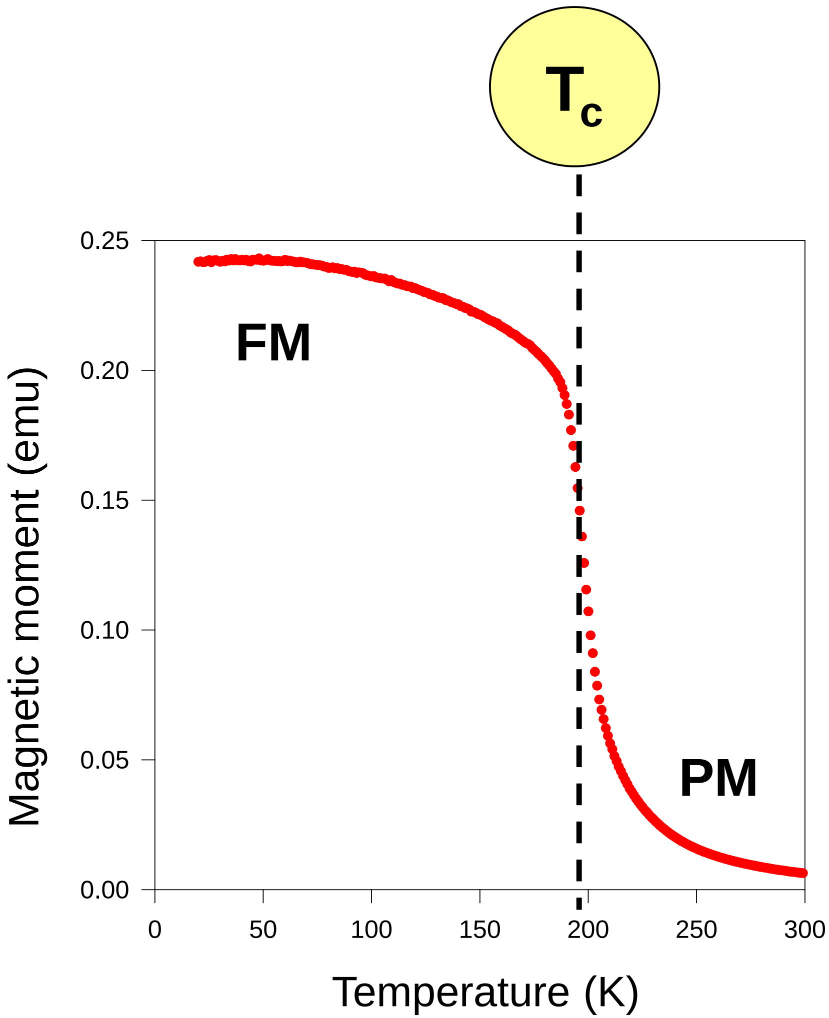
$La_{1-x}Ca_xMnO_3$



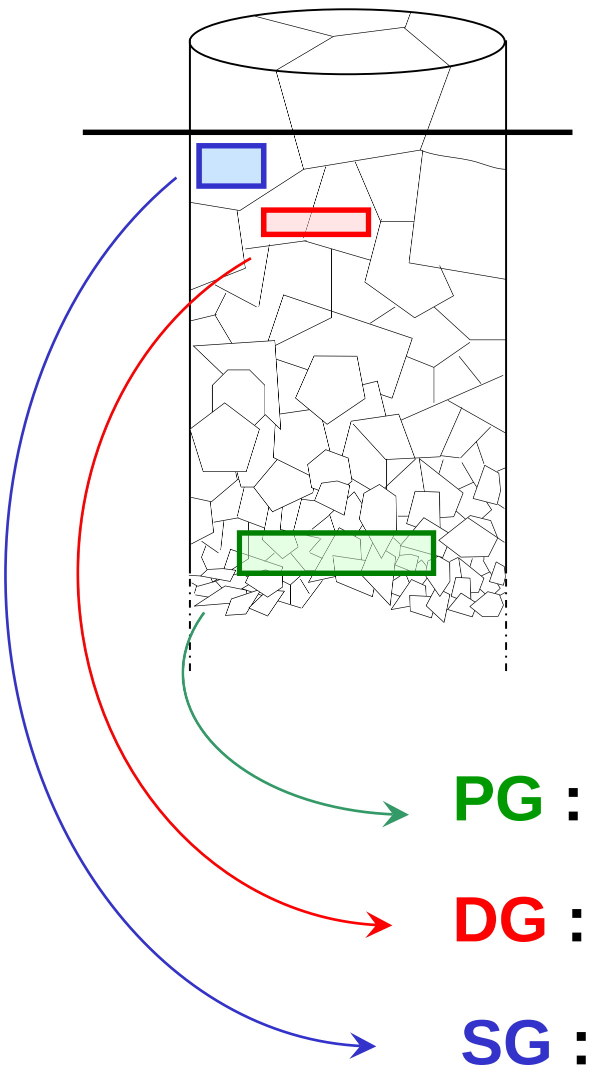


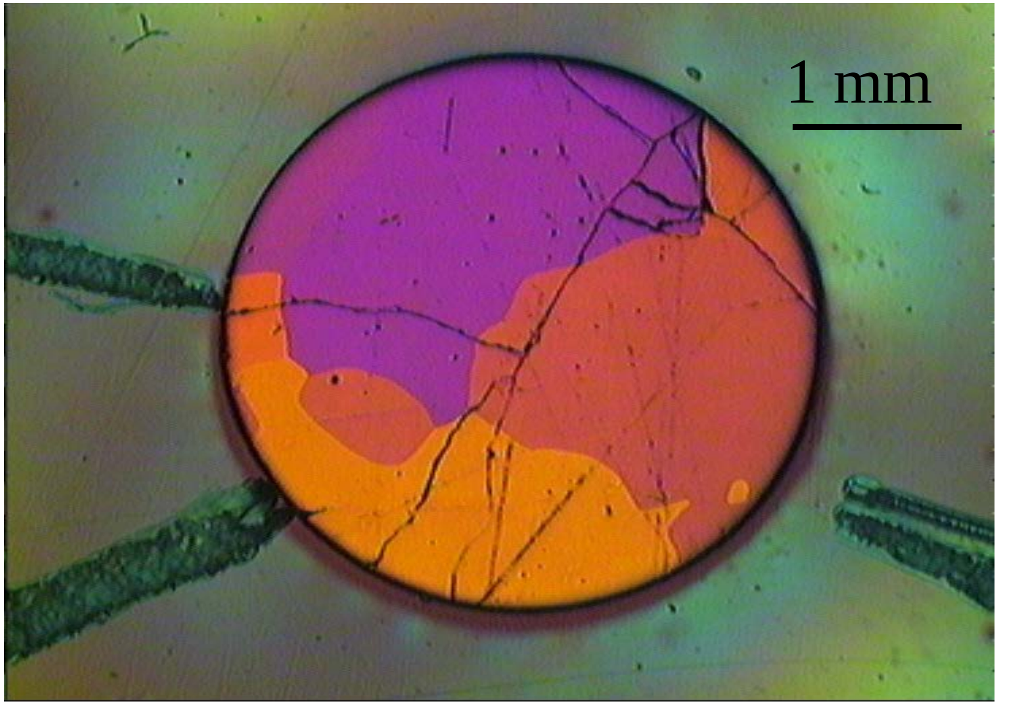
Metallic / Insulating transition ⟺ Ferromagnetic / Paramagnetic transition





Characterisation of the rod





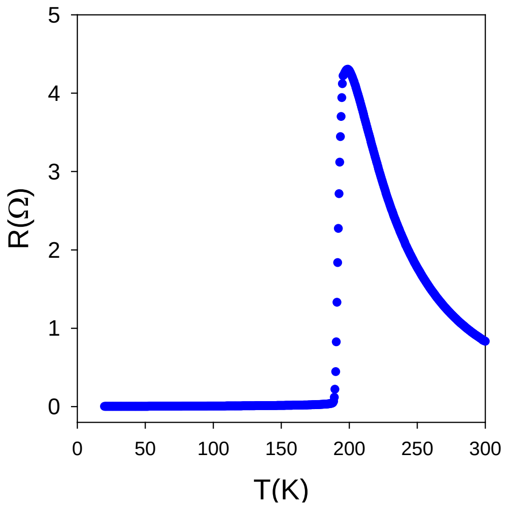
PG : **polygranular**

DG : **double grain**

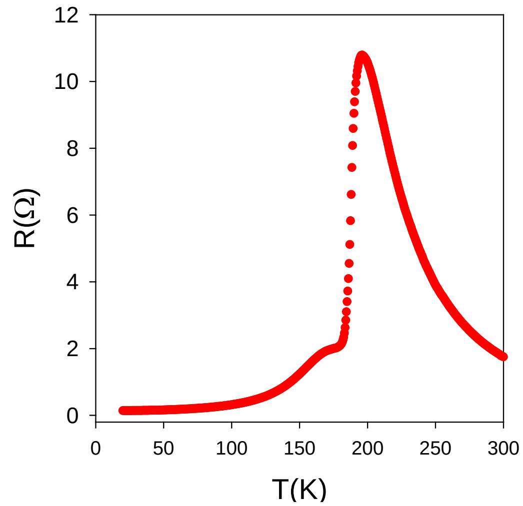
SG : **single grain**

Electrical resistance

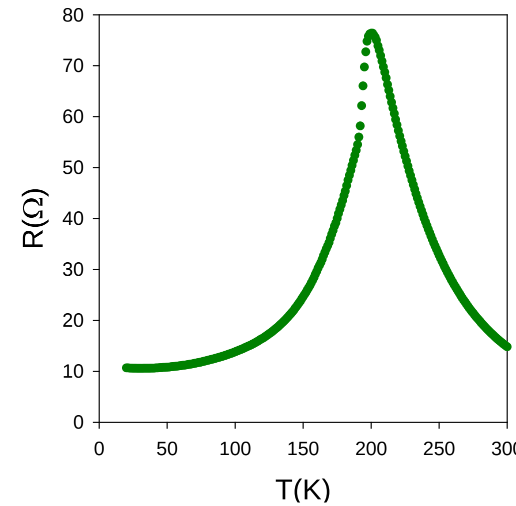
SG



DG



PG

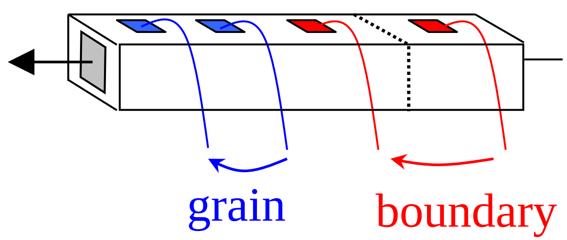


within a grain

↓

Sharp transition

across one
grain boundary



polygrain

↓

Broad
transition

Vanderbemden et al., Phys. Rev. B 68, 224418 (2003)

A single grain boundary in a bulk material

↓

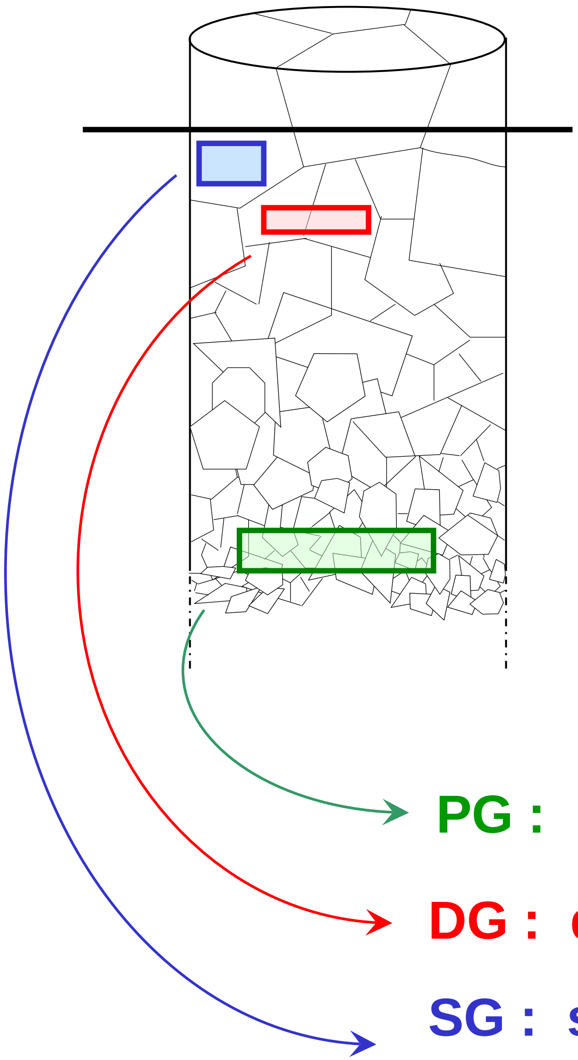
Significant effect on the electrical properties
(Low-field MR, Low-T MR, « foot »)

MR influenced by the magnetization of the adjacent grains

BUT …

Is there an influence on the magnetic properties ?

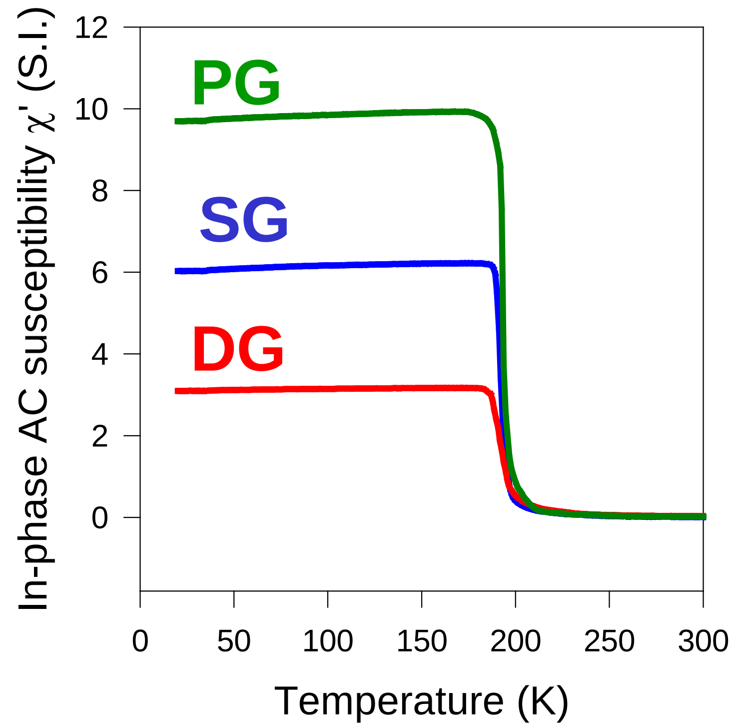
AC susceptibility



PG : polygrain

DG : double grain

SG : single grain

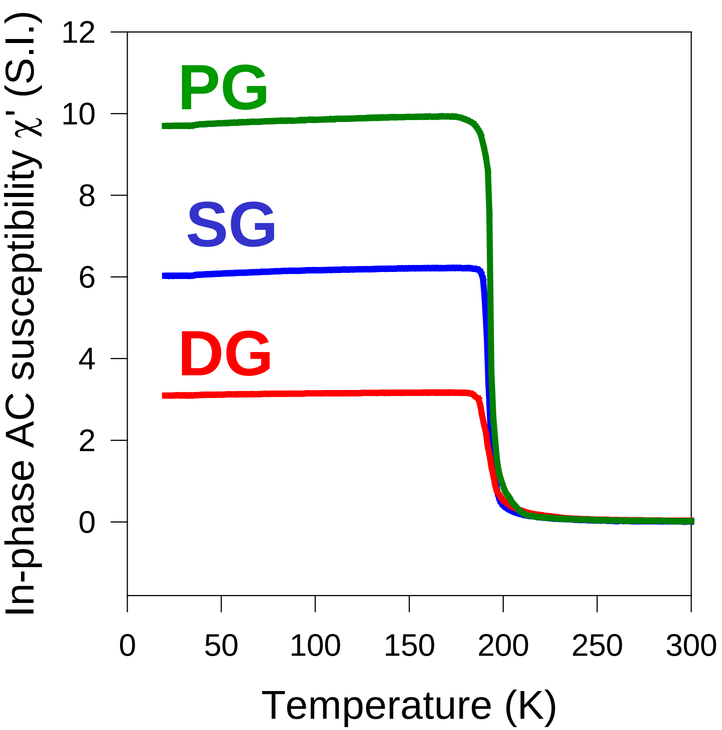


AC susceptibility

No logical order ???

→ Measurements influenced by
→
geometric effects

NOT by
microstructure effects !

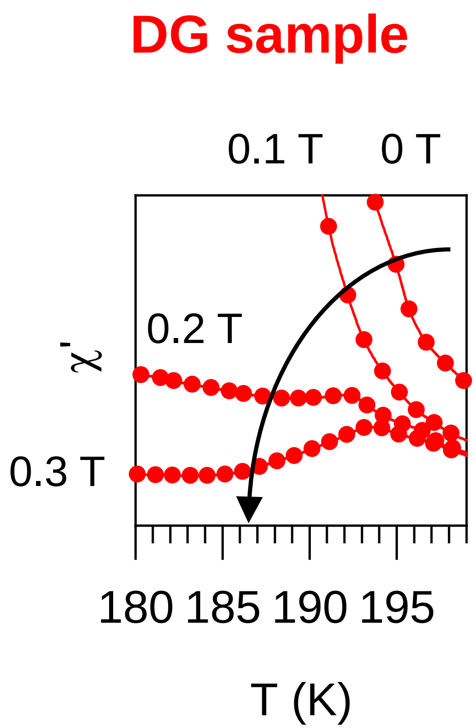


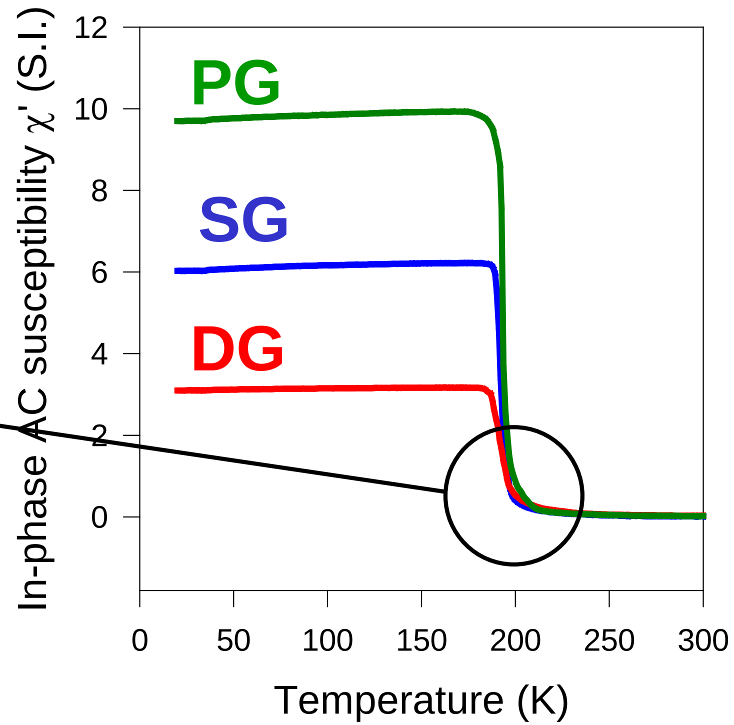
In the FM state, the AC susceptibility is given by

$$\chi \sim \frac{1}{D}$$

→ results linked to the demagnetization factor D for each particular sample

Influence of a superimposed DC field

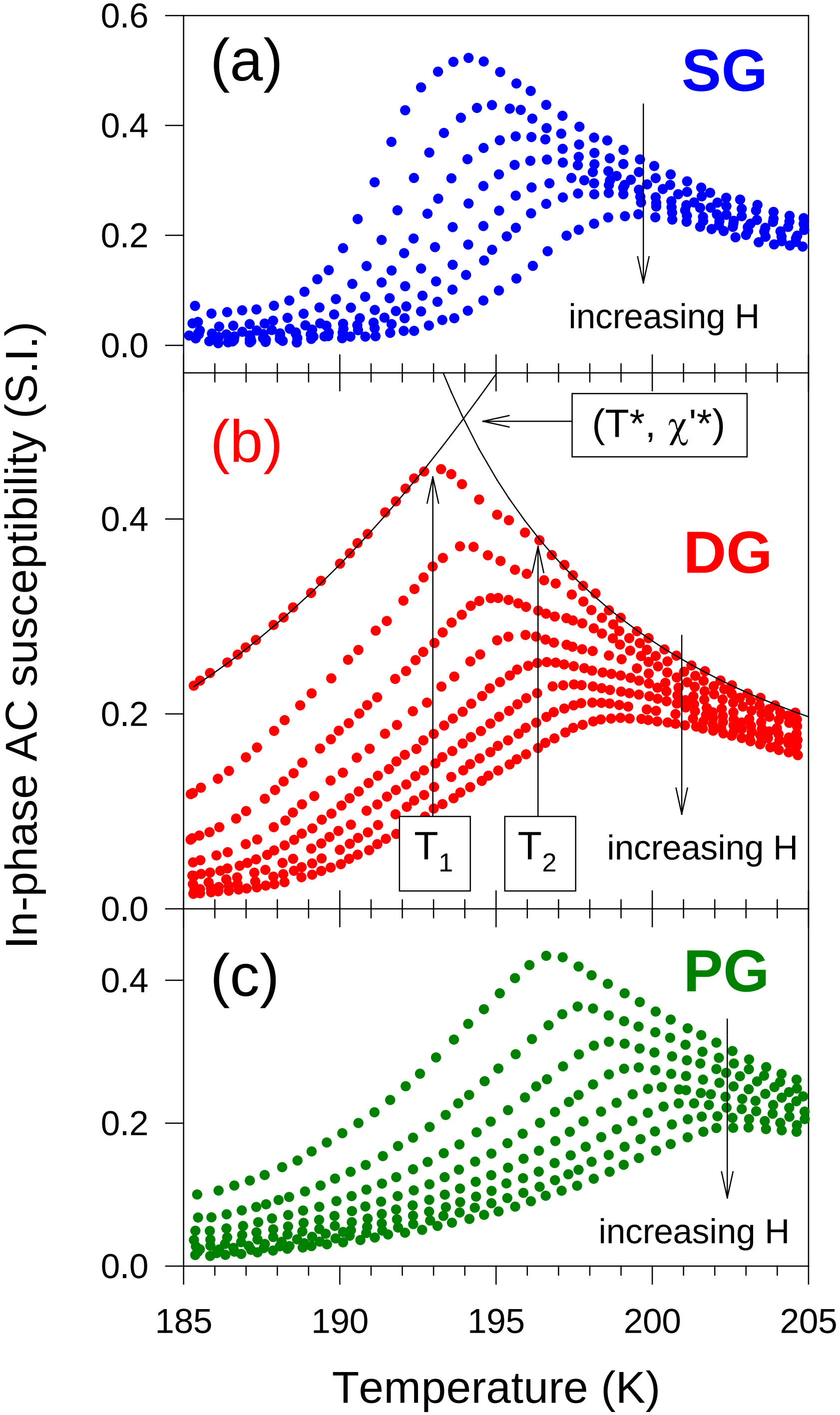




→ Suppression of the AC moment

→ Apparition of a <u>peak</u> related to critical fluctuations

Vanderbemden et al., Phys. Rev. B <u>68</u>, 224418 (2003)



$$H_i = H - D\,M$$

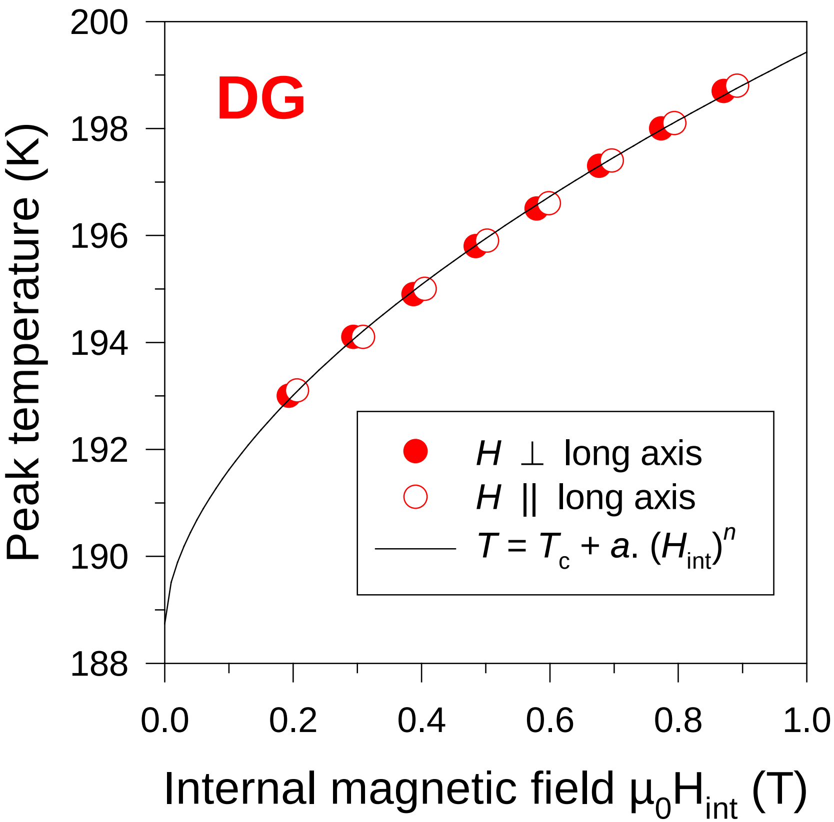
$$h = \frac{H_i}{T_c} \qquad t = \frac{T - T_c}{T_c}$$

$$T_{peak} = T_c + a(H_i)^n$$

$$n = \frac{1}{\beta + \gamma}$$

$$\chi_{peak} \sim h^{\left(\frac{1}{\delta} - 1\right)}$$

Critical exponents (β + γ) and δ



$$H_i = H - D\,M$$

$$T_{peak} = T_c + a(H_i)^n$$

Critical exponents

	$\beta + \gamma$	δ
SG	1.61	2.42
DG	1.49	2.54
PG	1.39	2.67

→ Unconsistent with 3D Heisenberg model (δ = 4.803)

→ Closer to the mean-field value (δ = 3)

→ Almost no effect of the microstructure

Double peak for the DG sample ?

Probably caused by a slight Tc difference between the two grains

Observable in AC measurements only in the presence of a **superimposed DC** field

↓

Powerful tool for revealing small Tc inhomogeneities and assess the sample quality !

Outline

- What are we measuring?
- How are we measuring?
- What kind of information can we extract?
- Beyond the classic setup : variants and particular designs

AC susceptometer for large AC fields

ELSEVIER

Cryogenics

Volume 34, Issue 10, October 1994, Pages 837-838

Paper

Variable temperature insert for a.c. susceptibility measurements at a.c. field amplitudes up to 0.1 T

(100 mT)

F. Gömöry, P. Lobotka, K. Fröhlich

INSTITUTE OF PHYSICS PUBLISHING — MEASUREMENT SCIENCE AND TECHNOLOGY

Meas. Sci. Technol. **15** (2004) 1195–1202 — PII: S0957-0233(04)74995-4

High-field ac susceptometer using Helmholtz coils as a magnetizer

D-X Chen

ICREA and Grup d'Electromagnetisme, Departament de Física, Universitat Autònoma de Barcelona, 08193 Bellaterra, Barcelona, Catalonia, Spain

The actual value of the maximum field is determined by the details of the magnetic measurements. In the ac susceptibility measurements at 77 K described in section 4, the maximum field amplitude is about 50 kA m^{-1} using the exemplified coils. This is a typical value for high-field ac susceptometers used in the high-temperature superconductor

(~ 62.5 mT)

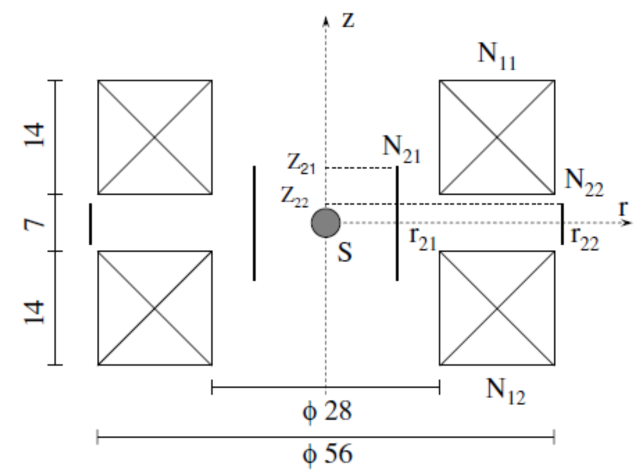


Figure 6. Axial cross-sectional view of the exemplified coil assembly. N_{11} and N_{12} are the magnetizing Helmholtz coils of square coil cross-section. N_{21} and N_{22} are the thin measuring and compensating coils. S is the measured sample. All the dimensions are in units of mm.

Gömöry F, Lobotka P and Fröhlich K (1994) *Cryogenics* 34 837-8

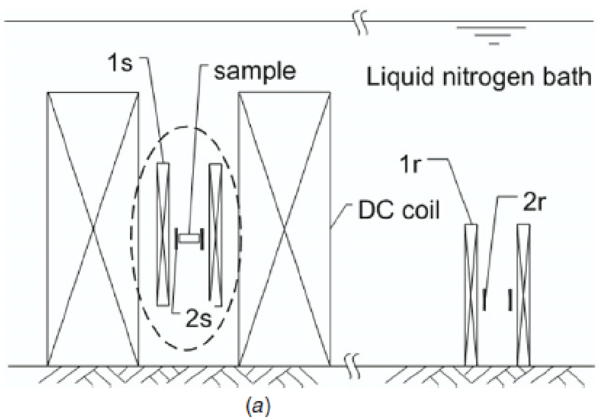
Chen D-X (2004) *Meas. Sci. Technol.* 15 1195-202

AC susceptometer for large samples

IOP Publishing | Measurement Science and Technology
Meas. Sci. Technol. 19 (2008) 085705 (10pp) | doi:10.1088/0957-0233/19/8/085705

An ac susceptometer for the characterization of large, bulk superconducting samples

P Laurent[1], J F Fagnard[1], B Vanderheyden[1], N Hari Babu[2], D A Cardwell[2], M Ausloos[3] and P Vanderbemden[1]



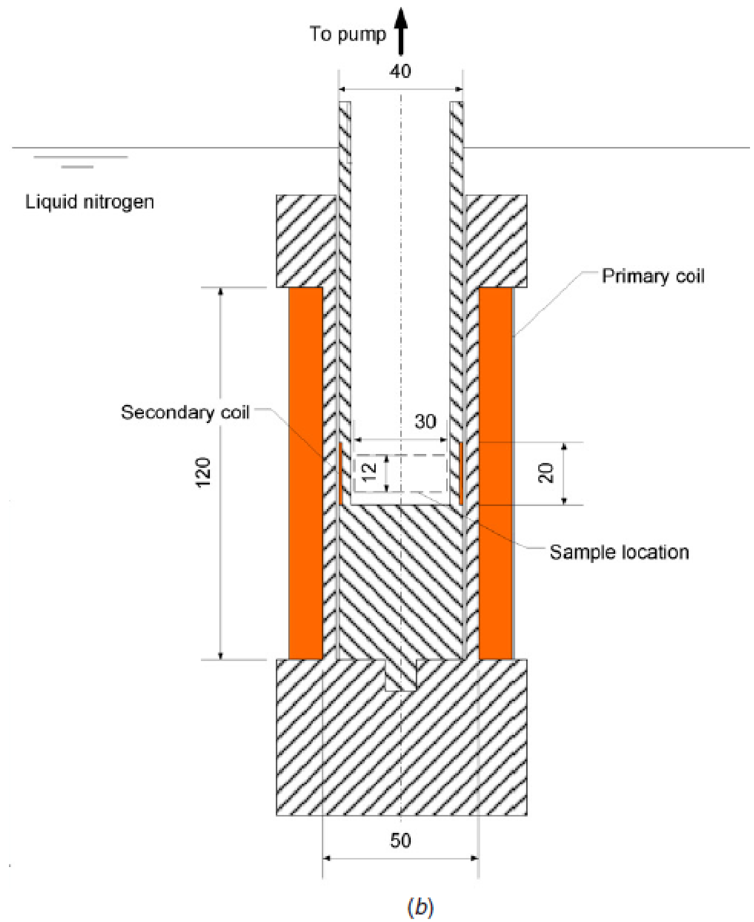
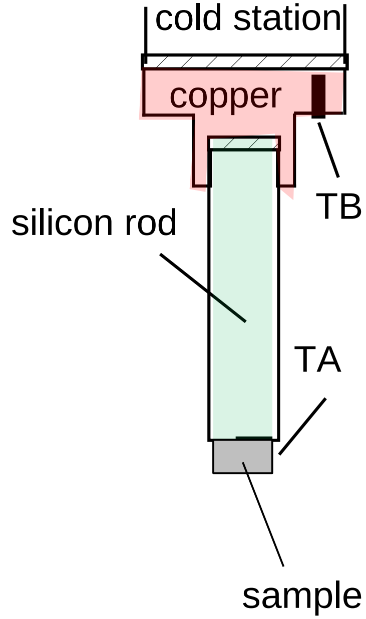


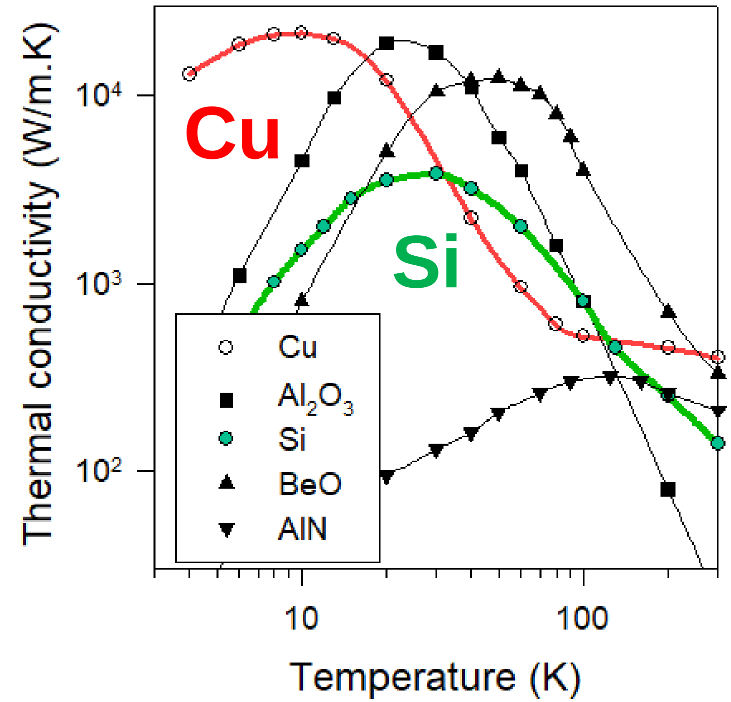
Figure 1. (*a*) Schematic illustration of the geometrical arrangement of the coils used for the ac susceptometer. The primary coil consists of two separate coils, one containing the sample (coil '1s') and one used for reference (coil '1r'). Coil '1s' can be inserted in a large coil that can generate a dc magnetic field. Primary coils '1s' and '1r' contain their own secondary coil, '2s' and '2r', respectively. (*b*) Cross-section of the part of the susceptometer containing the sample illustrating the geometry the primary coil '1s' and the secondary coil '2s'. All dimensions are given in mm. The sample, located in a vacuum vessel, is centred in the secondary coil '2s'. The reference coils ('1r' and '2r') are identical to those shown in figure 1(*b*).

P Laurent, J F Fagnard, B Vanderheyden, N Hari Babu, D A Cardwell, M Ausloos and P Vanderbemden, *Measurement Science and Technology* 19 (2008) 085705

AC susceptometer based on a cryocooler

- Requires a weakly electrically conducting sample holder with excellent thermal conductivity.
- A polycrystalline silicon (Si) rod proved to be a very good choice!





ELSEVIER

PII: S0011-2275(98)00063-0

Cryogenics **38** (1998) 839–842

Printed in Great Britain
0011-2275/98/$—see front matter

Design of an A.C. susceptometer based on a cryocooler

Ph. Vanderbemden

University of Liège, S.U.P.R.A.S., Montefiore Electricity Institute, B28, Sart-Tilman, B-4000 Liege, Belgium

Vanderbemden P *Cryogenics*, 38 839 (1998)

AC susceptometry at “high” f (> 10 kHz)

Available online at www.sciencedirect.com

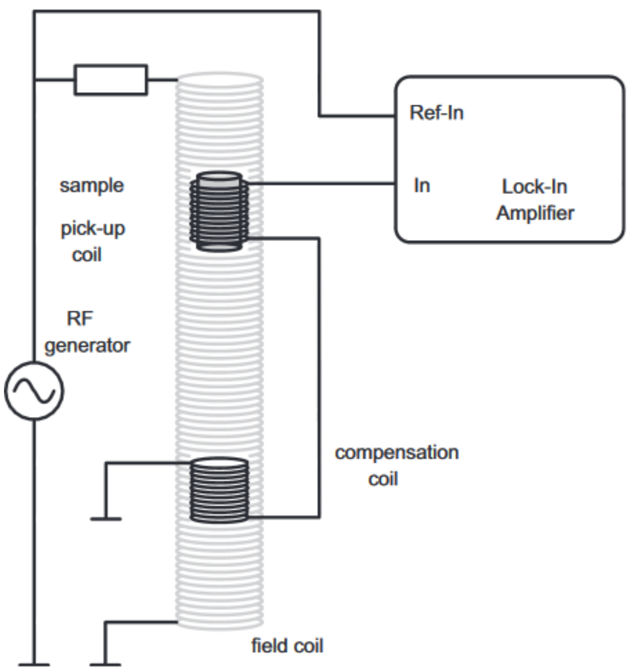
ScienceDirect

ELSEVIER

Journal of Magnetism and Magnetic Materials 311 (2007) 224–227

journal of magnetism and magnetic materials

www.elsevier.com/locate/jmmm



Metallic cobalt nanoparticles for heating applications

Matthias Zeisberger[a,*], Silvio Dutz[a], Robert Müller[a], Rudolf Hergt[a], Nina Matoussevitch[b], Helmut Bönnemann[b]

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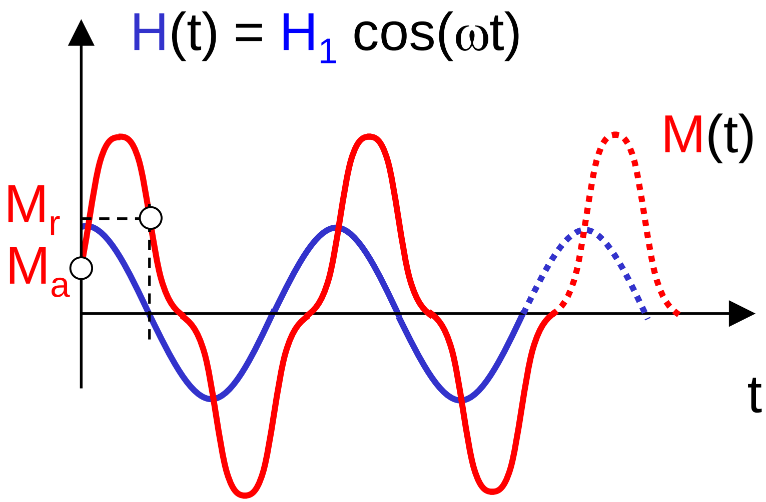
[b]*Forschungszentrum Karlsruhe, ITC-CPV, Post Box 3640, D-76021 Karlsruhe, Germany*

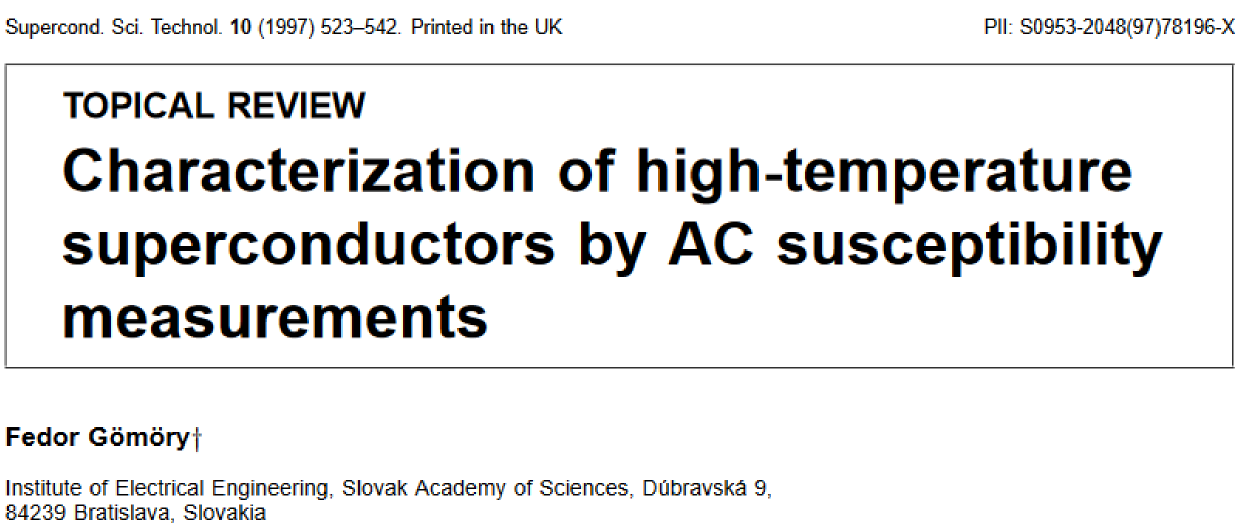
The field coil (∅ 14 mm × 75 mm, 75 turns) which is connected to a function generator provides an AC field with an amplitude up to 60 A/m and a frequency in the range from 20 Hz to 1 MHz. The sample is in a cylindric container (inner size ∅ 3 mm × 7 mm) which is placed inside the pick-up coil (25 turns). A compensation coil is used to cancel out the background signal.

"Wide-band" AC susceptometry



$$\chi_a = M_a / H_1 \qquad \chi_r = M_r / H_1$$

where M_a is the sample magnetization at the moment when the external field reaches the maximum: we can call it the 'amplitude magnetization'. M_r is the magnetization remaining in the sample at zero instantaneous value of the AC field: we can call it the 'remanent magnetization'



Supercond. Sci. Technol. **10** (1997) 523–542. Printed in the UK — PII: S0953-2048(97)78196-X

TOPICAL REVIEW

Characterization of high-temperature superconductors by AC susceptibility measurements

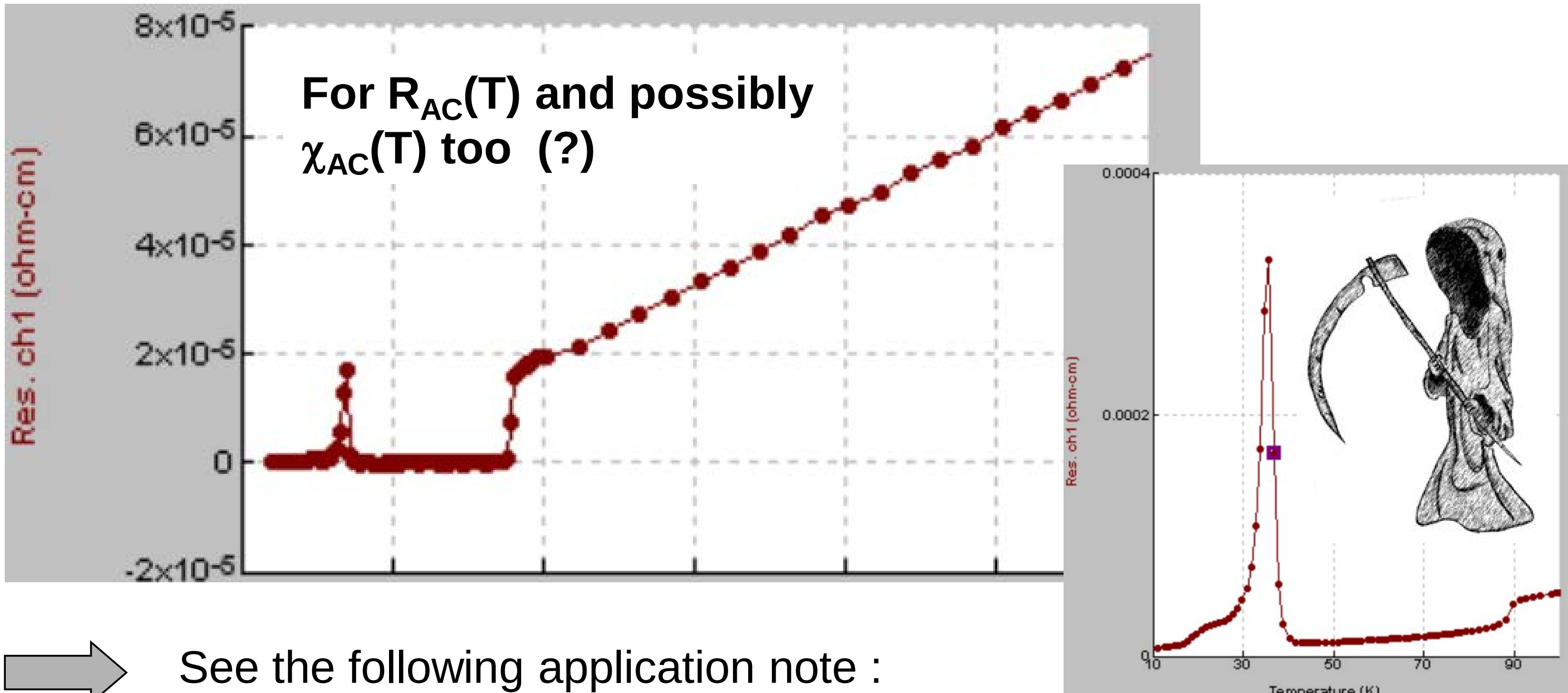
Fedor Gömöry†

Institute of Electrical Engineering, Slovak Academy of Sciences, Dúbravská 9, 84239 Bratislava, Slovakia

F. Gömöry, *Supercond. Sci. Technol.* **10** (1997) 523

! Parastic influence of the AC magnetic response of metals…

For $R_{AC}(T)$ and possibly $\chi_{AC}(T)$ too (?)



See the following application note :

Quantum Design

Distorted low-level signal readback of AC signals in the PPMS in the temperature range 25-35 K due to Inconel mitigation of inductive cross talk

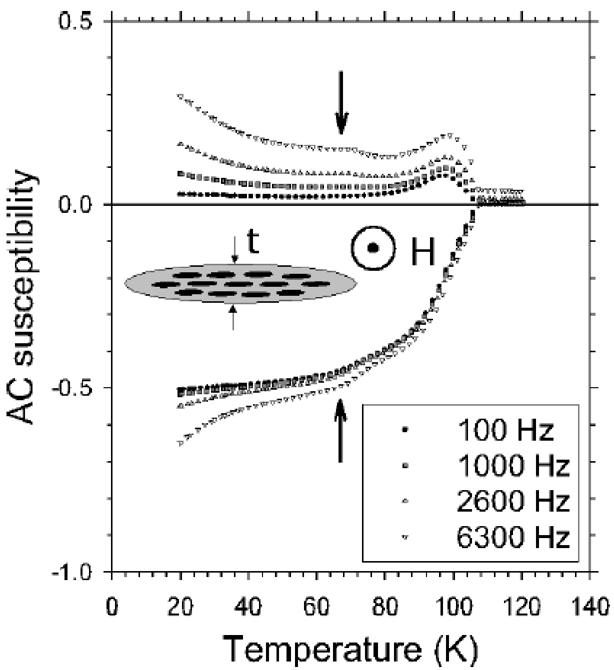
Example : AC susceptibility of **superconducting filaments** in a **silver matrix**

2976 IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 13, NO. 2, JUNE 2003

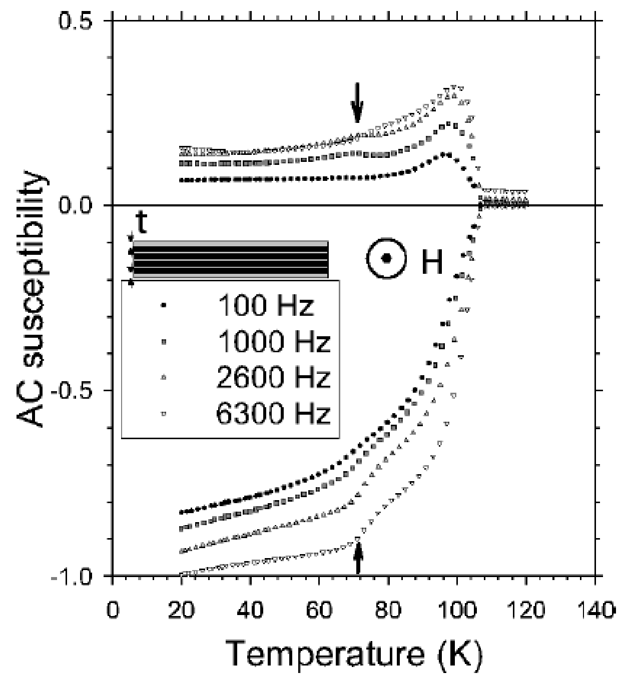
Anisotropic AC Behavior of Multifilamentary Bi-2223/Ag Tapes

J.-F. Fagnard, P. Vanderbemden, R. Cloots, and M. Ausloos

H_{AC} || filaments



$H_{AC} \perp$ filaments



J F Fagnard, P Vanderbemden, R Cloots and M Ausloos, *IEEE Trans. Appl. Supercond.* 13 2976 (2003)

For further information…

- See reference list on the following page
- See the old but excellent collection of papers
- See the following excellent introductory paper


American Journal of Physics

HOME BROWSE INFO FOR AUTHORS COLLECTIONS

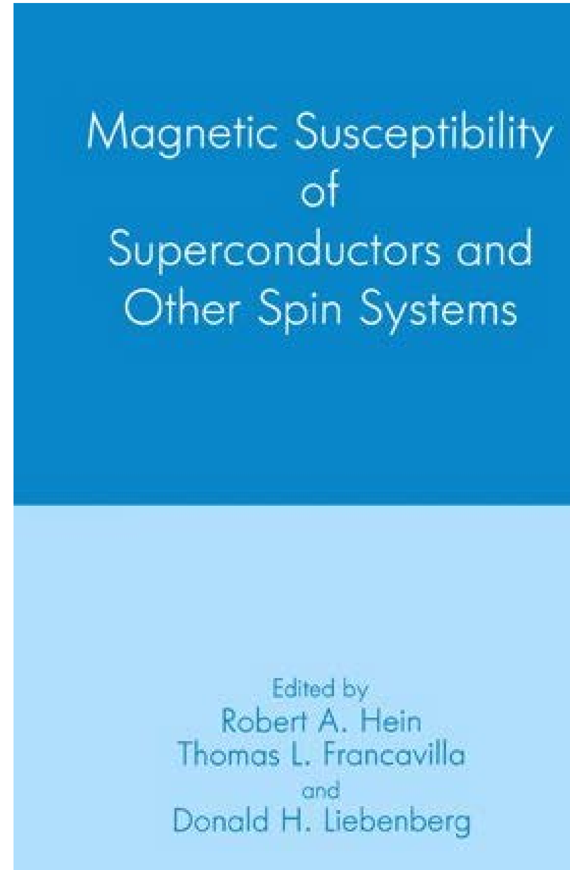
Home > American Journal of Physics > Volume 63, Issue 1 > 10.1119/1.17770

Published Online: 28 July 1998 Accepted: June 1994

Superconductivity: A guide to alternating current susceptibility measurements and alternating current susceptometer design

American Journal of Physics **63**, 57 (1995); https://doi.org/10.1119/1.17770

Martin Nikolo



Ed. Hein, R. A., Francavilla, T. L., Liebenberg, D.H. Plenum Press, New York. (1992)

Nikolo, M., Am. J. Phys., 63, 57 (1995)

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Contact us : Philippe.Vanderbemden@ulg.ac.be

Or visit us at :

LIÈGE université

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